



Physical Property Measurement System

Thermal Transport Option User's Manual

Part Number 1684-100B

Quantum Design

11578 Sorrento Valley Rd.
San Diego, CA 92121-1311
USA

Technical support (858) 481-4400
 (800) 289-6996
 Fax (858) 481-7410

Third edition of manual completed October 2002.

Trademarks

All product and company names appearing in this manual are trademarks or registered trademarks of their respective holders.

U.S. Patents

4,791,788 Method for Obtaining Improved Temperature Regulation When Using Liquid Helium Cooling
4,848,093 Apparatus and Method for Regulating Temperature in a Cryogenic Test Chamber
5,311,125 Magnetic Property Characterization System Employing a Single Sensing Coil Arrangement to Measure AC
 Susceptibility and DC Moment of a Sample (patent licensed from Lakeshore)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

C O N T E N T S

Table of Contents

PREFACE	vii
Contents and Conventions	vii
P.1 Introduction	vii
P.2 Scope of the Manual	vii
P.3 Contents of the Manual	vii
P.4 Conventions in the Manual	viii
 CHAPTER 1	
Introduction	1-1
1.1 Introduction	1-1
1.2 Overview of the Thermal Transport Option	1-1
1.2.1 Purpose of Measuring Thermal Transport Properties	1-3
1.3 Measurement Modes	1-3
1.3.1 Continuous Measurement Mode	1-3
1.3.2 Single Measurement Mode	1-3
1.4 Measured Thermal Properties	1-5
1.4.1 Thermal Conductivity	1-5
1.4.2 Seebeck Coefficient	1-5
1.4.3 Electrical Resistivity	1-6
1.4.4 Figure of Merit	1-6
1.5 Theory of Operation	1-7
1.5.1 Hardware	1-7
1.5.2 Thermal and Electrical Circuit	1-7
1.5.3 Software Models	1-8
1.5.4 Estimating Errors in the Data	1-9
1.5.5 Correcting for Heat Loss	1-11
1.5.6 Correcting for Seebeck Coefficient of Manganin Leads	1-11
1.6 Start-up Checklist for Secondary Installation	1-11
 CHAPTER 2	
Hardware	2-1
2.1 Introduction	2-1
2.2 Thermal Transport Hardware	2-1
2.2.1 Thermal Transport Sample Puck	2-2
2.2.2 User's Kit	2-3
2.2.3 Nickel Calibration Samples	2-5
2.2.4 WaveROM EPROM	2-5
2.2.4.1 Replacing the WaveROM Chip	2-6
2.2.5 Thermal Transport Connection Cable	2-6
2.2.6 User Bridge Board	2-7
2.3 ACT Hardware	2-7
2.3.1 Model 7100 AC Transport Controller	2-7
2.3.2 AC Board	2-7

2.4 High-Vacuum Hardware.....	2-8
2.4.1 Contact Baffle.....	2-8
2.5 Calibrating New Shoe Assemblies	2-9

CHAPTER 3

Software.....	3-1
3.1 Introduction	3-1
3.2 Overview of Thermal Transport Software.....	3-1
3.2.1 Measurement Units.....	3-2
3.3 Thermal Transport Control Center	3-3
3.3.1 Control Center Tabs.....	3-3
3.3.2 Measurement Menu	3-5
3.3.2.1 Options for Advanced Users.....	3-8
3.3.3 System Status.....	3-9
3.4 Thermal Transport Data Files	3-10
3.4.1 Saving Raw Thermal Transport Data	3-10
3.4.2 Data File Header	3-10
3.4.3 Format of Measurement Data Files.....	3-11
3.4.4 Format of Raw Data Files	3-13
3.5 Data Examination	3-14

CHAPTER 4

Sample Preparation	4-1
4.1 Introduction	4-1
4.2 Sample-Mounting Considerations	4-1
4.2.1 Geometry	4-2
4.2.2 Lead-Mounting Epoxies	4-3
4.2.2.1 Silver-Filled H20E Epoxy	4-3
4.2.2.2 Tra-Bond 816H01 Epoxy.....	4-3
4.3 Two-Probe and Four-Probe Lead Configurations.....	4-4
4.3.1 Two-Probe Lead Configuration.....	4-4
4.3.2 Four-Probe Lead Configuration.....	4-5
4.4 Checking the Sample Contact.....	4-6
4.5 Using the Puck-Mounting Station.....	4-6

CHAPTER 5

Measurements	5-1
5.1 Introduction	5-1
5.2 Taking Thermal Transport Measurements.....	5-1
5.2.1 Connect Leads to the Sample.....	5-2
5.2.2 Measure the Sample Dimensions.....	5-2
5.2.3 Mount the Sample.....	5-3
5.2.4 Install the Sample	5-3
5.2.5 Start the High-Vacuum System	5-3
5.2.6 Open the Data File	5-4
5.2.7 Define the Measurement.....	5-4
5.2.8 Run the Measurement	5-5
5.2.8.1 Running the Measurement Interactively	5-5
5.2.8.2 Running the Measurement in a Sequence	5-5
5.2.9 Scanning or Ramping the Temperature While Measuring.....	5-5

5.3 Measurement Mode Parameters.....	5-6
5.3.1 Continuous Measurement Mode.....	5-6
5.3.2 Single Measurement Mode.....	5-8
5.4 Description of Measurement Process.....	5-9

CHAPTER 6

Troubleshooting	6-1
6.1 Introduction	6-1
6.2 Jumps or Noise in the Data	6-1
6.2.1 Gaps in the Data Versus Temperature	6-2
6.2.2 Steps in the Data	6-2
6.3 Thermal Radiation “Tail” in the Thermal Conductivity Data.....	6-3
6.4 High-Vacuum Problems	6-3

CHAPTER 7

Maintenance	7-1
7.1 Introduction	7-1
7.2 Using the Puck Adjustment Tool.....	7-1
7.3 Greasing the Puck Fingers and the Coldfoot Clamp.....	7-2

APPENDIX A

Installation	A-1
A.1 Introduction.....	A-1
A.2 Installing Thermal Transport Hardware.....	A-1
A.3 Installing Thermal Transport Software	A-3

APPENDIX B

Status Codes and Error Messages	B-1
B.1 Introduction.....	B-1
B.2 System Status Codes	B-1
B.2.1 General PPMS System Status Codes	B-1
B.2.2 Thermal Transport System Status Codes	B-3

APPENDIX C

Pinout Tables	C-1
C.1 Introduction.....	C-1
C.2 Thermal Transport Pinouts.....	C-1
C.2.1 Sample Connections.....	C-1

References	References-1
-------------------------	--------------

Index	Index-1
--------------------	---------

Figures

Figure 1-1. Thermal and Electrical Connections for an Idealized Sample	1-7
Figure 1-2. Heat Pulse and Temperature and Voltage Response at Hot and Cold Thermometer Shoes in an Idealized Sample	1-8
Figure 2-1. TTO Puck with Radiation Shield	2-2
Figure 2-2. Puck-Mounting Station with Puck	2-3
Figure 2-3. Thermal Transport Option User's Kit	2-4
Figure 2-4. WaveROM EPROM on AC Board in Model 6000 PPMS Controller	2-5
Figure 2-5. Thermal Transport Connection Cable	2-6
Figure 2-6. Front Panel on Model 7100 AC Transport Controller	2-7
Figure 2-7. Baffle Assembly with Contact Baffle	2-8
Figure 2-8. Close-up View of Contact Fingers and Charcoal Holder on Contact Baffle Assembly	2-8
Figure 2-9. Calibration Fixture Plugged into TTO Puck and Illustrating Sockets for Each Shoe Assembly	2-10
Figure 2-10. Thermal Transport Calibrate Thermometers and Heater Wizard	2-10
Figure 3-1. Thermal Transport Log Window	3-2
Figure 3-2. Control Center Install Tab	3-3
Figure 3-3. Control Center Data File Tab	3-3
Figure 3-4. Control Center Sample Tab	3-4
Figure 3-5. Control Center Waveform Tab	3-4
Figure 3-6. Control Center Advanced Tab	3-5
Figure 3-7. Settings Tab in Thermal Transport Measurement Dialog Box	3-6
Figure 3-8. Thermal Tab in Thermal Transport Measurement Dialog Box	3-6
Figure 3-9. Resistivity Tab in Thermal Transport Measurement Dialog Box	3-7
Figure 3-10. Mode Tab in Thermal Transport Measurement Dialog Box	3-8
Figure 3-11. Advanced Tab in Thermal Transport Measurement Dialog Box	3-8
Figure 3-12. Error Count Dialog Box	3-14
Figure 4-1. Examples of Leads Mounted in Two-Probe Configuration	4-4
Figure 4-2. Example of Leads Mounted in Four-Probe Configuration	4-5
Figure 5-1. Thermal Tab in Thermal Transport Measurement Dialog Box	5-6
Figure 5-2. Resistivity Tab in Thermal Transport Measurement Dialog Box	5-8
Figure 7-1. Puck Adjustment Tool	7-1
Figure A-1. Thermal Transport Option Connection Diagram	A-2
Figure C-1. Illustration of TTO Sample Connections, Showing Hardware Ports	C-2
Figure C-2. Top View of Pinout of Connector Sockets on Thermal Transport Sample Puck	C-3

Tables

Table 1-1. System Requirements for the Thermal Transport System.....	1-2
Table 1-2. Thermal Transport System Parameters	1-2
Table 1-3. Thermal Transport System Components	1-2
Table 1-4. Styles for Measurements Taken in Single Measurement Mode	1-4
Table 2-1. Recommended Sample Parameters for Nickel Calibration Samples.....	2-5
Table 3-1. PPMS System Data Items That Can Be Saved to the TTO Measurement Data File	3-9
Table 3-2. Fields in Thermal Transport Measurement Data File.....	3-11
Table 3-3. Fields in Thermal Transport Raw File.....	3-13
Table 4-1. Sample Geometries and Range of Measurable Thermal Conductivities	4-2
Table 4-2. Approximate Thermal Conductance of Epoxies.....	4-5
Table 5-1. Minimum and Maximum Parameter Limits for Continuous Mode Measurements	5-6
Table 5-2. General Settings for Continuous Mode Measurements	5-7
Table 5-3. Resistivity Excitation Parameters for Continuous Mode Measurements	5-8
Table B-1. Status Associated with Bits of General System Status Field	B-1
Table B-2. TTO Status Codes.....	B-3
Table C-1. TTO Sample Connections.....	C-2

Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.

P.2 Scope of the Manual

This manual discusses the Thermal Transport option (TTO) for the Physical Property Measurement System (PPMS). This manual explains how to use the TTO system and it explains the theory of operation for TTO. This manual describes the hardware and software that are unique to TTO, and it includes maintenance and troubleshooting information.


For detailed information about the PPMS MultiVu software, which is the parent software application running the PPMS, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

P.3 Contents of the Manual

- Chapter 1 presents an overview of the TTO system and of the TTO theory of operation.
- Chapter 2 discusses and illustrates the hardware used with TTO.
- Chapter 3 discusses the TTO software and TTO data files.
- Chapter 4 explains how to prepare samples for TTO measurements.
- Chapter 5 explains how to take measurements with TTO and describes the measurement process.
- Chapter 6 contains troubleshooting suggestions.
- Chapter 7 explains basic maintenance procedures.

- Appendix A explains how to install the TTO hardware and software.
- Appendix B contains status codes and error messages.
- Appendix C contains pinout tables.

P.4 Conventions in the Manual

File menu	Bold text distinguishes the names of menus, options, buttons, and panels appearing on the PC monitor or on the Model 6000 PPMS Controller LCD screen.
File>Open	The ➤ symbol indicates that you select multiple, nested software options.
STATUS	Bold text and all CAPITAL letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.
.dat	The <i>Courier</i> font distinguishes characters you enter from the PC keyboard or from the Model 6000 PPMS Controller front panel. The <i>Courier</i> font also distinguishes code and the names of files and directories.
<Enter>	Angle brackets < > distinguish the names of keys located on the PC keyboard.
<Alt+Enter>	A plus sign + connecting the names of two or more keys distinguishes keys you press simultaneously.
 NOTE	A pointing hand and the word NOTE introduce a supplementary note.
CAUTION!	Cautionary notes are preceded with the word CAUTION! This signals conditions that could result in loss of information or damage to your equipment.
WARNING!	Warnings are preceded with the word WARNING! This signals conditions that could result in bodily harm or loss of life.

Introduction

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the TTO system.
- Section 1.3 describes the TTO system measurement modes.
- Section 1.4 explains how the TTO system measures thermal properties.
- Section 1.5 explains the TTO system's theory of operation.
- Section 1.6 contains the start-up checklist for secondary installation of the TTO.

1.2 Overview of the Thermal Transport Option

The Quantum Design Thermal Transport option (TTO) for the Physical Property Measurement System (PPMS) enables measurements of thermal properties, including thermal conductivity κ and Seebeck coefficient (also called the thermopower) α , for sample materials over the entire temperature and magnetic field range of the PPMS. The TTO system measures thermal conductivity, or the ability of a material to conduct heat, by monitoring the temperature drop along the sample as a known amount of heat passes through the sample. TTO measures the thermoelectric Seebeck effect as an electrical voltage drop that accompanies a temperature drop across certain materials. The TTO system can perform these two measurements simultaneously by monitoring both the temperature and voltage drop across a sample as a heat pulse is applied to one end. The system can also measure electrical resistivity ρ by using the standard four-probe resistivity provided by the PPMS AC Transport Measurement System (ACT) option (Model P600). All three measurement types are essential in order to assess the so-called “thermoelectric figure of merit,” $ZT = \alpha^2 T / \kappa \rho$, which is the quantity of main interest if you are investigating thermoelectric materials.

While the measurements taken with the TTO system are quite elementary in principle, they have eluded commercialization because the data was typically very error prone, time consuming, and laborious, due—for example—to problems in controlling heat flow and accurately measuring small temperature differentials in a convenient manner. The TTO system has solved or greatly reduced many of these experimental complications. TTO uses convenient sample mounting, small and highly accurate Cernox chip thermometers, and sophisticated software that dynamically models an AC heat flow through the sample and corrects for any heat losses that occur. The PPMS with the High-Vacuum option (Model P640) provides an ideal environment for the custom-designed TTO sample puck, and the ACT option (Model P600) powers the sample heater and takes resistivity measurements. Table 1-1 on the following page lists the TTO system requirements.

Table 1-1. System Requirements for the Thermal Transport System *

COMPONENT	FUNCTION
PPMS Resistivity Option (Model P400)	Provides user bridge board that reads two thermometer shoes.
PPMS AC Transport Measurement System (Model P600)	Outputs current to heater and sample while providing low-noise, phase-sensitive detection.
PPMS High-Vacuum Option (Model P640)	Provides thermal isolation for measurements. Cryopump or Turbo Pump may be used.
PPMS MultiVu Software Version 1.1.6 or Later	Provides single user interface for PPMS and PPMS options.

* In addition to the requirements in Table 1-1, the PPMS Continuous Low-Temperature Control (CLTC) option (Model P800) is highly recommended. CLTC provides extended low-temperature control.

Table 1-2. Thermal Transport System Parameters

PARAMETER	VALUE
Pressure	High vacuum ($\sim 10^{-4}$ torr)
Temperature	1.9–390 K
Magnetic field	0–14 T when $T > 20$ K

If you require use of significant magnetic fields ($H > 0.1$ T) at temperatures below $T \sim 20$ K, please inquire with Quantum Design.

Table 1-3. Thermal Transport System Components

COMPONENT	PART NUMBER	ILLUSTRATION
Thermal Transport sample puck including Isothermal radiation shield	4084-570 4084-575 4084-579	Figure 2-1
Two plug-in thermometer shoes	4084-580T	Figure 2-1
Plug-in heater shoe	4084-585	Figure 2-1
User's kit	4084-569	Figure 2-3
Two nickel standard samples	4084-593	Figure 2-3
WaveROM EPROM for AC board	3084-043	Figure 2-4
Thermal Transport connection cable	3084-582	Figure 2-5
Thermal Transport software module		

1.2.1 Purpose of Measuring Thermal Transport Properties

In measuring the thermal transport properties of a material specimen—such as thermal conductivity κ and Seebeck coefficient α —a researcher can learn considerable information about the electronic as well as the ionic lattice structure of that specimen. Thermal conductivity is a measure of the ability of a material to conduct heat, so measuring this quantity provides information about scattering of heat-carrying phonons and electrons. The Seebeck coefficient describes the thermal diffusion of free charge carriers (electrons or holes), which creates an electric field inside a material when a temperature gradient is sustained. Much like the electrical resistivity, this property is very sensitive to subtle changes in the electronic scattering processes and can be a powerful probe in that regard.

Taken together with electrical resistivity ρ , the thermal conductivity and Seebeck coefficient also provide a measure of the so-called thermoelectric figure of merit $Z = \alpha^2/(\kappa\rho)$, which is a quantity of practical significance because it quantifies a material's ability to transport heat by the application of an electric current (Peltier effect), or conversely, a material's ability to generate an electric field by passing a thermal current (Seebeck effect, described above). The figure of merit is usually expressed as the dimensionless quantity $Z \times T$, where $Z \times T \sim 1$ is a common benchmark for viability of a material for thermoelectric applications.

1.3 Measurement Modes

The TTO system includes two measurement modes:

- Continuous measurement mode
- Single measurement mode

All properties measurements offered by TTO can be performed in either of these two modes. Parameters for each measurement mode (see Section 5.3) may be specified prior to running any measurement in that mode.

1.3.1 Continuous Measurement Mode

In continuous measurement mode, measurements are being taken continually and the adaptive software is adjusting parameters (such as heater power and period) to optimize the measurements. This mode is amenable to slow sweeps of system variables such as temperature or magnetic field, and it is often the most rapid way of obtaining data because you do not have to wait for the system to reach equilibrium before measuring. The continuous mode is also expedited by the use of a sophisticated curve-fitting algorithm that determines the steady-state thermal properties by extrapolating from the response to a relatively short (typically several minutes) heat pulse.

1.3.2 Single Measurement Mode

The single measurement mode is slower than continuous measurement mode because it requires that the system reach a steady state in both the heater “off” and “on” states, which also implies that temperature or field slewing is unavailable. The advantage of single measurement mode is that no

subtle curve-fitting calculations are required, so interpretation of the raw data is in principle more straightforward. Researchers who study thermal transport properties usually employ this steady-state technique because of its simplicity and robustness. In either style of single measurement—stability or timed—described below in Table 1-4, data is first taken in the heater “off” state once the system settles. After the user-specified heater power is applied, the system waits for the selected equilibrium condition before making the final measurement in the heater “on” state. You can view the live ΔT vs. time data in the **Waveform** tab of the Thermal Transport control center to monitor measurement progress.

Table 1-4. Styles for Measurements Taken in Single Measurement Mode

MEASUREMENT STYLE	DEFINITION
Stability	System takes first measurement in heater “off” state once temperature stability at both hot and cold sample thermometers is within a specified window, stated either as a percentage of T or as an absolute number (in kelvin). After heat is applied, system waits for the same stability criterion to be met before taking final measurement. Heater power is turned off after conclusion of this measurement. User-specified timeout forces system to take a measurement at timeout period even if stability criterion has not been met.
Timed	Sends heat pulse of user-specified duration into sample. System takes a measurement of temperatures and thermal voltages before applying heat, and then takes final measurement at end of heat pulse.

1.4 Measured Thermal Properties

The TTO system is set up to measure four thermal transport properties:

- Thermal conductivity
- Seebeck coefficient
- Electrical resistivity
- Thermoelectric figure of merit

If thermal conductivity, Seebeck coefficient, and electrical resistivity are all measured, then the thermoelectric figure of merit, which is the algebraic combination of these three measurements, can be determined.

Separate measurement protocols are provided for thermal conductivity, Seebeck coefficient, and electrical resistivity because these individual quantities may be more accurately measured by using excitation currents and temperature differentials optimized for each situation. Limits for the parameters defining each measurement may be specified prior to running the measurement. Section 5.3 discusses the measurement parameters.

Each measured thermal transport property may be determined in either of the two measurement modes (continuous or single) supported by the TTO system; refer to Section 1.3. You select a measurement mode, and then you select the thermal properties to measure in that mode.

1.4.1 Thermal Conductivity

The TTO system measures thermal conductivity κ by applying heat from the heater shoe in order to create a user-specified temperature differential between the two thermometer shoes. The TTO system dynamically models the thermal response of the sample to the low-frequency, square-wave heat pulse, thus expediting data acquisition. TTO can then calculate thermal conductivity directly from the applied heater power, resulting ΔT , and sample geometry.

1.4.2 Seebeck Coefficient

The TTO system determines the Seebeck coefficient (also called the thermopower) α by creating a specified temperature drop between the two thermometer shoes—just as it does to measure thermal conductivity. However, for Seebeck coefficient the voltage drop created between the thermometer shoes is also monitored. The additional voltage-sense leads on these thermometer shoes are connected to the ultra-low-noise preamplifier of the ACT system.

1.4.3 Electrical Resistivity

The TTO system measures electrical resistivity ρ by using a precision DSP current source and phase-sensitive voltage detection. The specifications for this AC resistivity measurement are essentially identical to those for the AC Transport Measurement System (ACT) option, because the same high-performance hardware is used by both TTO and ACT. The *Physical Property Measurement System: AC Transport Option User's Manual* discusses the ACT measurements in detail.

1.4.4 Figure of Merit

The dimensionless thermoelectric figure of merit ZT is determined here simply as the algebraic combination $ZT = \alpha^2 T / \kappa \rho$ of the three measured quantities—thermal conductivity, Seebeck coefficient, and electrical resistivity—discussed above.

1.5 Theory of Operation

Benefits of the design of the TTO system include the following:

- Four-terminal geometry minimizes the effects of thermal and electrical resistance of the leads
- Continuous measurements while slewing in temperature provide high density of data
- Careful attention to the removal of effects of temperature drift, thermal radiation, and other systematic errors
- Robust, easy-to-use, fully automated measurements

1.5.1 Hardware

When measuring in continuous mode, the DSP hardware in the Model 7100 AC Transport Controller generates the heat pulse in the chip resistor heater on the sample, which can be described as an “on” cycle of constant power followed by an “off” cycle of equal duration. The waveform for this pulse was programmed specially for the TTO system in the waveROM EPROM on the AC board, so older AC boards must have the old waveROM swapped for the new waveROM (labeled “THRMXPT 4201”) to run TTO. Section 2.2.4 discusses the waveROM EPROM in more detail.

Figure 1-2 on the following page illustrates the heat pulse as well as the temperature and voltage response at the hot and cold thermometer shoes in an idealized sample.

1.5.2 Thermal and Electrical Circuit

The thermal and electrical connections for an idealized TTO sample are shown in Figure 1-1. For clarity, the sample is shown mounted in the four-probe geometry. The four basic physical elements are illustrated: the sample, the epoxy bonds that adhere the leads to the sample, the copper leads, and the heater and thermometer shoe assemblies that screw down onto the leads. For thermal conductivity and Seebeck coefficient measurements, heat is applied to one end of the sample by running current through the heater ($Q_{+/-}$). The temperatures T_{hot} and T_{cold} are measured at the thermometer shoes. Also during the heat pulse, the Seebeck voltage ($\Delta V = V_+ - V_-$) is monitored. Heat exits the sample to the coldfoot. Time traces of ΔT and ΔV during the heat pulse are illustrated in Figure 1-2.

Electrical resistivity measurements are made both before and after the heat pulse described above. Current ($I_{+/-}$) flows through the sample and the voltage drop across the sample is monitored using the $V_{+/-}$ leads.

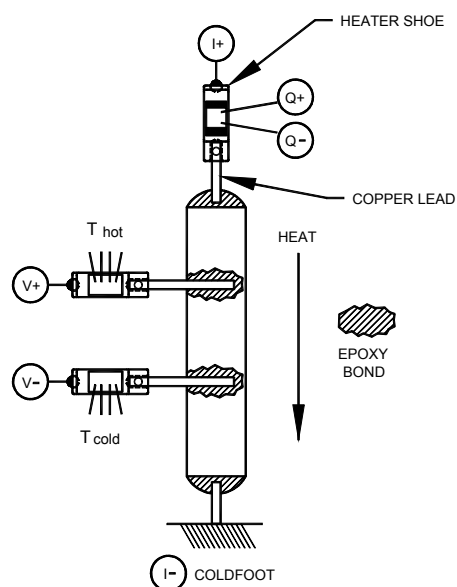


Figure 1-1. Thermal and Electrical Connections for an Idealized Sample

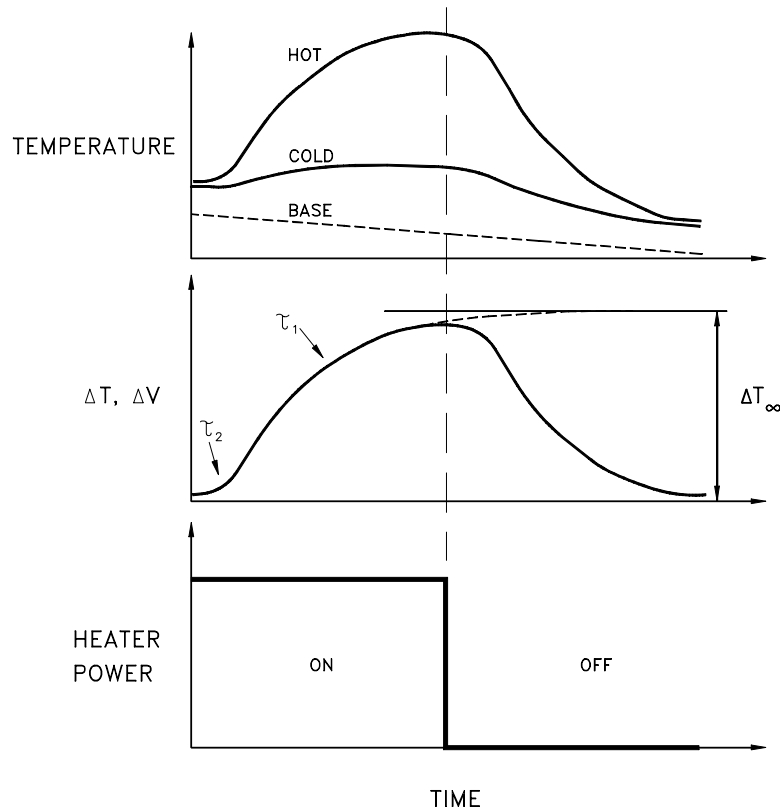


Figure 1-2. Heat Pulse and Temperature and Voltage Response at Hot and Cold Thermometer Shoes in an Idealized Sample

- Top panel: Time trace of hot and cold thermometers during an idealized heat pulse; note that the PPMS base temperature is slewing.
- Middle panel: Corresponding temperature ΔT and voltage ΔV differentials across the sample, indicating thermal time constants τ_1 and τ_2 and the estimate of the asymptotic differential ΔT_∞ .
- Bottom panel: Heater power during square-wave heat pulse.

1.5.3 Software Models

In continuous measurement mode (Section 1.3.1), the software uses adaptive algorithms to optimize measurement parameters such as heater current, heat pulse period, and resistivity excitation amplitude and frequency. Once the ΔT vs. time data over the duration of the heat pulse is obtained, a nonlinear least-squares fitting routine, which fits the data to the empirical formula, is launched:

$$\Delta T_{\text{model}} = \Delta T_\infty \times \{1 - [\tau_1 \times \exp(-t/\tau_1) - \tau_2 \times \exp(-t/\tau_2)]/(\tau_1 - \tau_2)\} \quad (\text{Equation 1-1})$$

where ΔT_∞ represents the asymptotic temperature drop across the sample if the heater is left on indefinitely, and τ_1 and τ_2 are long and short empirical time constants, respectively, for the sample (see Figure 1-2). The fitting routine performs an exhaustive search over the space of these three parameters, reducing the space iteratively until the parameter values that yield the minimum in the residual of the curve fit are identified satisfactorily. Equation 1-1 is appropriate to the data taken during the heating pulse, while the data taken during the cooling pulse is simultaneously fit essentially by changing the sign of the model equation: $\Delta T_{\text{model,cooling}} = A - \Delta T_{\text{model,heating}}$, where A is a constant. Due to long

thermal diffusion times (τ_1), the thermal history of the sample must be accounted for in the model, and this is achieved by including the remanent effects of the two previous pulses in modeling the current pulse.

The fitting routine for Seebeck coefficient data is similar, yet it is less computationally intensive. The ΔV vs. time data is read back from the DSP buffer at the end of the measurement, and after the ΔT vs. time data is fit to obtain τ_1 and τ_2 , a linear least-squares routine fits the data to the following equation:

$$\Delta V_{\text{model}} = \Delta V_{\infty} \times \{1 - [\tau_1 \times \exp(-t/\tau_1) \pm \tau_2' \times \exp(-t/\tau_2')]/(\tau_1 - \tau_2')\} + bt + c \quad (\text{Equation 1-2})$$

where ΔV_{∞} is the asymptotic Seebeck voltage drop akin to ΔT_{∞} in equation 1-1, b and c are parameters that describe linear drift and offset voltages, respectively, and $\tau_2' = 0 \dots \tau_1$ is swept so that for each value of τ_2' a linear regression in ΔV_{∞} , b , and c is performed. Note that “ \pm ” is used between the exponential terms and signifies that a full search is done for each sign. The physical significance of this is that the Seebeck coefficient of the material responsible for the short time constant τ_2 (that is, the leads) may be of the opposite sign as that for the material associated with the long time constant (that is, the sample). This is in contrast to the case of the thermal conductivity, which is always positive. The parameter for the linear voltage drift b is included here to account for varying thermal voltages in the wiring to the sample and also the slow microvolt-level drift in the preamp electronics.

A similar measurement technique, previously published by Maldonado¹, describes modeling of the thermal and thermoelectric response of a sample to a low-frequency, square-wave heat pulse. However, the thermal circuit considered in that work was considerably simpler than that which is appropriate to TTO, and hence the modeling was done differently.

1.5.4 Estimating Errors in the Data

The software also estimates the standard deviations (σ) in the reported quantities of thermal conductivity, Seebeck coefficient, electrical resistivity, and figure of merit ZT . This is done by estimating the goodness of the curve fits to κ , α , and ρ by calculating the residual of the curve fit. We make the assumption that this residual reflects the error in our estimate of the quantity (ΔT or ΔV), and this is true when the data deviates from the curve fit in a random manner. If deviations are systematic, as can be seen by inspecting the data in the `.raw` file, this indicates that the curve fit does not properly represent the data and the quantity and that error estimates are incorrect. If this occurs, consult Chapter 6 for troubleshooting tips. The residual for the ΔT vs. time curve fitting is calculated as follows:

$$\text{Residual} = R_{\Delta T} = \sqrt{\frac{\sum_i (\Delta T_i - \Delta T_{i,\text{model}})^2}{N}} \quad (\text{Equation 1-3})$$

where N is the number of data points making up the curve. In the measurements of κ and α , $N = 64$, while for ρ , $N = 128$. Since the thermal conductance $K = P/\Delta T$, errors in the heater power P (see the next section) must also be taken into account. The standard deviation in the conductivity is then calculated:

$$\sigma(\kappa) = \kappa \times \sqrt{\left(\frac{R_{\Delta T}}{\Delta T_{\infty}}\right)^2 + \left(\frac{2IR\delta I}{P}\right)^2 + \left(\frac{0.2 \times P_{\text{loss}}}{P}\right)^2 + \left(\frac{0.1 \times T_{\infty} \times K_{\text{shoes}}}{P}\right)^2} \quad (\text{Equation 1-4})$$

¹ Maldonado, O. Pulse method for simultaneous measurement of electric thermopower and heat conductivity at low temperatures. *Cryogenics*, vol. 32, (no. 10), 1992. 908–12.

The first term is the residual of the curve fit mentioned above, the second term propagates the error in the heater current I (heater resistance is R) due to the digital-analog converter, the third term is the error in the estimation of the sample radiation term where 20% combined error in the estimation of sample surface area and emissivity is assumed, and the last term is the error in the thermal conductance leak from the shoe assemblies K_{shoes} where a 10% error in this correction is assumed (see the next section for details on heat losses).

The error in the measurement of the thermal voltage ΔV vs. time has a similar expression as equation 1-3, so the standard deviation in the Seebeck coefficient $\alpha = \Delta V / \Delta T$ is the following:

$$\sigma(\alpha) = \alpha \times \sqrt{\left(\frac{R_{\Delta V}}{\Delta V_{\infty}}\right)^2 + \left(\frac{R_{\Delta T}}{\Delta T_{\infty}}\right)^2} \quad (\text{Equation 1-5})$$

Resistivity measurements are made both preceding and following each thermal measurement so that the average of the two ρ and $\sigma(\rho)$ values is reported in the data file. The residual of the curve fits R_p is obtained from the stream of voltage V vs. time data as the following:

$$\text{Residual} = R_p = \sqrt{\frac{\sum_i (V_i - V_{i,\text{model}})^2}{N}} \quad (\text{Equation 1-6})$$

and the standard deviation is calculated simply as

$$\sigma(\rho) = \rho \times \frac{R_p}{V_{pp}} \quad (\text{Equation 1-7})$$

where V_{pp} is the peak-to-peak amplitude of the voltage vs. time signal.

The standard deviation in the figure of merit ZT is obtained by propagating the errors from each of the measurements:

$$\sigma(ZT) = ZT \times \sqrt{\left(\frac{2\sigma(\alpha)}{\alpha}\right)^2 + \left(\frac{\sigma(\kappa)}{\kappa}\right)^2 + \left(\frac{\sigma(\rho)}{\rho}\right)^2 + \left(\frac{\sigma(T)}{T}\right)^2} \quad (\text{Equation 1-8})$$

where the last term is the standard deviation of the sample temperature over the measurement.

1.5.5 Correcting for Heat Loss

Thermal conductance is determined as $K = P/\Delta T$ where P is the heat flowing through the sample. Since the heat flux cannot be measured directly, the net conducted heat through the sample is estimated as the power (I^2R) dissipated in the heater resistor, minus losses due to radiation or thermal conduction down the leads from the shoe assemblies. Thus the conductance is determined as follows:

$$K \text{ [W/K]} = (I^2R - P_{\text{rad}}) / \Delta T - K_{\text{shoes}} \quad (\text{Equation 1-9})$$

where

$$K_{\text{shoes}} = aT + bT^2 + cT^3 \quad (\text{Equation 1-10})$$

is a standard estimate of the thermal conductance of the shoe assemblies (a , b , and c are constants), and

$$P_{\text{rad}} = \sigma_T \times (S/2) \times \varepsilon \times (T_{\text{hot}}^4 - T_{\text{cold}}^4) \quad (\text{Equation 1-11})$$

is the radiation from the sample, S is the total sample surface area, ε is the infrared emissivity of the radiating surface (see Section 5.2.2 for more information on estimating the emissivity), $T_{\text{hot/cold}}$ are the average temperatures of the hot and cold thermometers during the measurement, and $\sigma_T = 5.67 \times 10^{-8} \text{ W m}^{-2} \text{ K}^{-4}$ is the Stefan-Boltzmann constant. The factor of $1/2$ in the equation is due to the approximation that only half of the sample surface area is radiating at the hot temperature, while the other half is at the cold temperature.

Since radiative heat losses are often very difficult to accurately estimate, you should expect errors in measurements of thermal conductance above $T \sim 300 \text{ K}$ that are on the order of $\pm 1 \text{ mW/K}$.

1.5.6 Correcting for Seebeck Coefficient of Manganin Leads

The manganin leads that connect the shoes to the connector plugs have a small Seebeck coefficient (no more than $\sim 1 \text{ } \mu\text{V/K}$ at any temperature), and this has been estimated and subtracted from the “Seebeck Coef. ($\mu\text{V/K}$)” data column in the data file. However, the column “Seebeck Volt. (μV)” is uncorrected.

1.6 Start-up Checklist for Secondary Installation

1. Verify that the new AC board with the waveROM EPROM is installed in the Model 6000 PPMS Controller. Refer to Sections 2.2.4 and 2.2.4.1.
2. Verify that the Resistivity option (Model P400) is installed and the user bridge board is in the Model 6000 PPMS Controller.
3. Verify that all proper connections are made between the Model 6000 PPMS Controller and the Model 7100 AC Transport Controller. Refer to Figure A-1.
4. Verify that the High-Vacuum option (Model P640) is installed and activated.
5. Verify that the gray Lemo cable for the Thermal Transport option is properly connected.
6. Verify that the TTO software is installed as a PPMS MultiVu option.

Hardware

2.1 Introduction

This chapter contains the following information:

- Section 2.2 discusses and illustrates the TTO hardware.
- Section 2.3 discusses the ACT option hardware that is used with TTO.
- Section 2.4 discusses the High-Vacuum option hardware that is used with TTO.
- Section 2.5 explains how to calibrate new shoe assemblies.

2.2 Thermal Transport Hardware

The TTO hardware includes the following:

- Thermal Transport sample puck
- Isothermal radiation shield (tube and cap)
- Two calibrated plug-in thermometer shoes
- One calibrated plug-in heater shoe
- Three uncalibrated shoes: two thermometer shoes and one heater shoe
- User's kit
- Two nickel calibration samples
- Thermal Transport connection cable

The TTO system also uses ACT hardware (Section 2.3), High-Vacuum hardware (Section 2.4), and, if required, an AC board ROM upgrade kit.

2.2.1 Thermal Transport Sample Puck

The Thermal Transport sample puck (Figure 2-1) plugs into the 12-pin socket at the bottom of the PPMS sample chamber. The Thermal Transport puck is inserted into the sample chamber by using the standard PPMS puck extraction tool (part number 4084-110). All 12 pins on the puck are used for thermal transport measurements (Appendix C lists pinouts). The puck serial number is written on the plastic socket of the base.

Modularized shoe assemblies, including two temperature/voltage shoes and one heater/current shoe, on the Thermal Transport puck connect to the three five-pin sockets on the green printed circuit board. Each gold-plated copper “shoe” has a hole in which the appropriate sample lead is inserted and held in the shoe by a small stainless steel metric M1 screw. The temperature/voltage shoe assemblies contain a Cernox 1050 thermometer as well as a voltage lead that is soldered to the shoe itself. The heater/current shoe assembly contains a resistive heater chip as well as an electrical current source lead (I+) that is soldered to the shoe. At the other end of each shoe assembly is a five-pin electrical plug on which the serial number is written. Each shoe type—heater or thermometer—is individually serialized. The 2-inch-long, 0.003-inch-diameter wires used for leads on the shoe assemblies are designed to minimize thermal conduction from the sample to the puck, and hence all are made of manganin alloy with the exception of the current (I+) lead, which is made of PD-135 low-resistance copper alloy. Two Sharpie permanent markers, red and blue, have been included with the TTO system so that you can color the alumina Cernox chip housing on each thermometer shoe, as well as the plastic electrical plug at the other end, to indicate the hot and cold probes. Marking the housing is convenient because it is easy to confuse the two sets of wires between the sockets and the shoes. The sides of the sockets for the thermometer shoe assemblies are painted so that the middle socket is red (hot probe) and the left-side socket is blue (cold probe). The right-hand socket, which is unpainted, is used only by the heater shoe assembly.

The sample is connected to the puck at the coldfoot, which contains a Phillips screw and a stainless steel clamp on the bottom that clamps onto the sample lead. This is the thermal sink for the sample, so good thermal contact is important here. If achieving good thermal contact is a concern, a small amount of Apiezon H Grease, which is included in the TTO user’s kit (Section 2.2.2), can be used on the sample lead to improve contact to the coldfoot. Note that good electrical contact is also required at the coldfoot if resistivity measurements are being made.

The copper isothermal radiation shield screws into the base of the puck and is designed to minimize radiation between the sample and the environment. The cap is removable so that you can verify that the leads and the sample do not touch the shield. A copper shield plate is also placed between the sample stage and the PC board sockets to minimize radiation effects.

CAUTION! Use care when threading the radiation shield onto the puck. The copper metal is soft, so excessive force or misthreading of the piece can easily damage the threads.

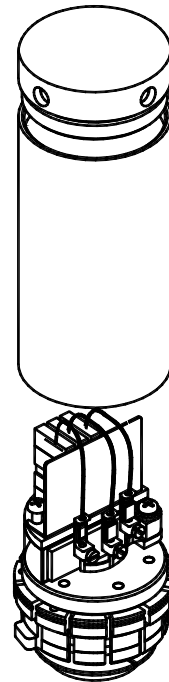


Figure 2-1. TTO Puck with Radiation Shield

2.2.2 User's Kit

The user's kit contains miscellaneous hardware and consumables that are needed for mounting leads on samples as well as calibrating the spare shoe assemblies provided with the Thermal Transport option. The convenient portable toolbox (see Figure 2-3 on the following page) helps keep the items in the kit organized.

The contents of the user's kit include the following.

- **Puck-mounting station**

A pivoting, rotating socket is mounted to a heavy base (Figure 2-2) and holds the Thermal Transport sample puck in a fixed position, giving you better access to the sample leads while you are connecting the shoes or making other adjustments. Tighten the two thumbscrews on the mounting station once the desired orientation for the puck is achieved.

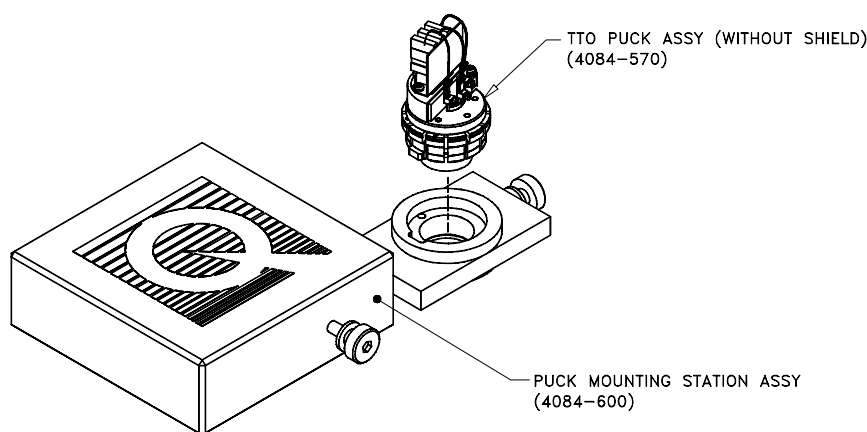


Figure 2-2. Puck-Mounting Station with Puck

- **Nickel calibration samples**

Two “comb-shaped” samples made of nickel are used as standards for all measured thermal transport properties. Refer to Section 2.2.3 for more information on the nickel samples.

- **Gold-plated copper samples**

Two similar comb-shaped samples of copper are provided as thermal shunts to help provide an isothermal environment while calibrating shoe assemblies.

- **Calibration fixture**

This fixture is used in conjunction with calibration software and allows the heater and thermometer shoes to be calibrated.

- **Consumable items**

These items consist of a sampler of gold-plated copper leads, including both wires and disks; a sample of two types of epoxies for mounting leads; and a small tube of Apiezon H Grease to increase thermal contact between the leads and shoes or the coldfoot. Chapter 4 contains more detailed information about mounting leads to samples.

- **User tools**

These tools include small slotted and Phillips screwdrivers for sample mounting, and an extractor tool to remove the electrical connector plug ends of the shoe assemblies from the puck. This extractor tool holds the connector plug by sliding into the grooves on each side of the plug.

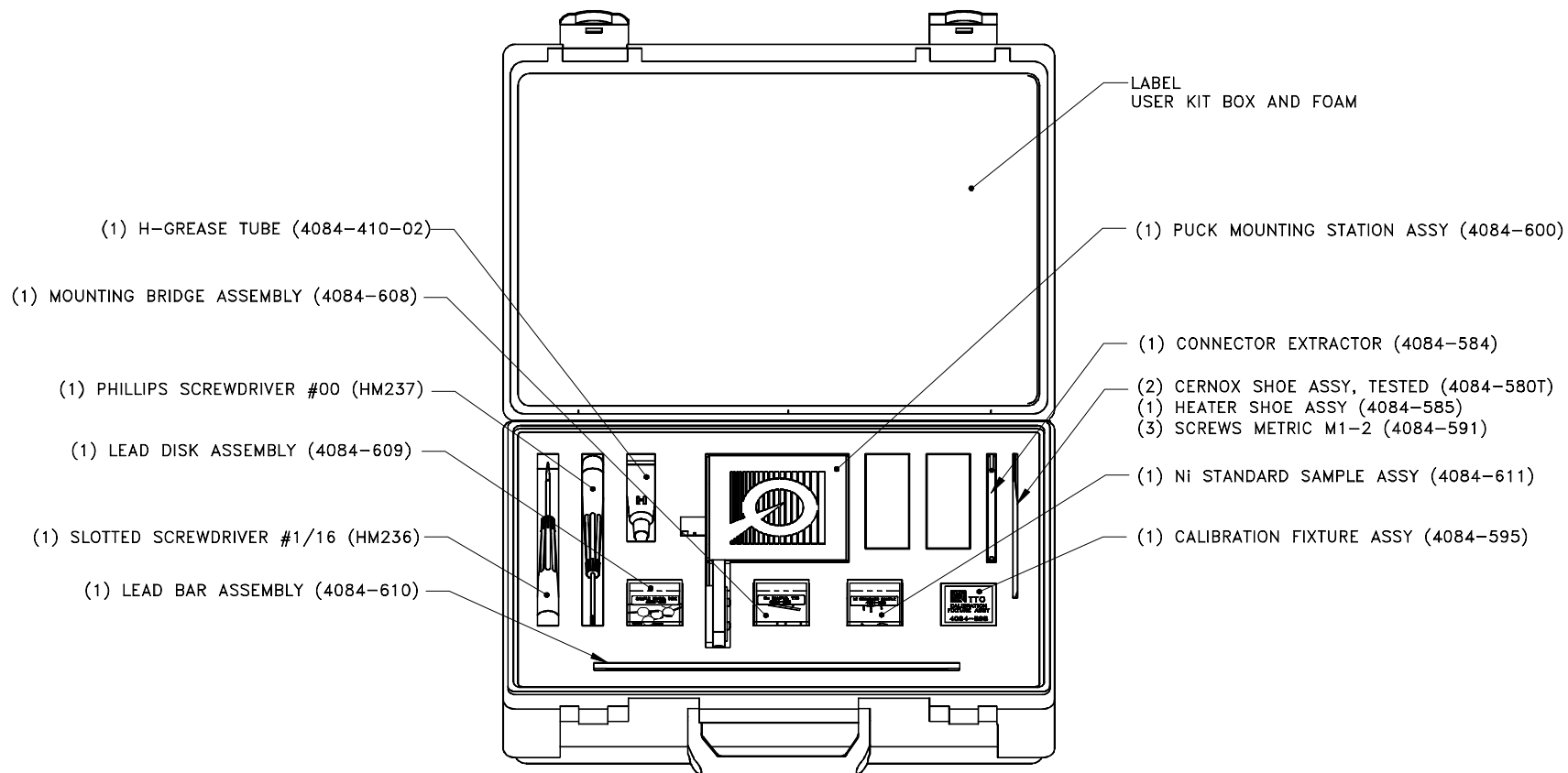


Figure 2-3. Thermal Transport Option User's Kit

2.2.3 Nickel Calibration Samples

Two samples of nickel metal (grade 201) are supplied in the user's kit in the form of thin plates stamped in a four-probe "comb" configuration (see Section 4.3) and can be used as references and calibration verification for all standard thermal transport measurements: thermal conductivity κ , Seebeck coefficient α , and electrical resistivity ρ . Variations in the geometrical factor A/l on the order of $\pm 5\%$ are to be expected from sample to sample, which will be reflected in the κ and ρ data. Table 2-1 lists standard dimensions for the Ni standard. It is also recommended that the Ni standard be mounted on the puck in the vertical configuration in order to avoid touching the radiation shield.

Table 2-1. Recommended Sample Parameters for Nickel Calibration Samples

PARAMETER	VALUE
Cross-sectional area	0.32 mm ²
Length	8.3 mm
Surface area	35 mm ²
Emissivity	0.5

2.2.4 WaveROM EPROM

The DSP board that is used with the ACMS and ACT options contains a square ROM chip that holds waveform tables for the excitation current generated by the DSP. The TTO system requires that a new waveform table—that of a square-wave pulse—be added to this library. All new AC boards include this new waveROM chip, but some customers with older boards must install the new chip in order to use the TTO system. If you are one of these customers, a new waveROM and a PLCC chip extraction tool (AC board ROM upgrade kit) have been sent to you so that you can swap out the old chip for the new one. Refer to Section 2.2.4.1.

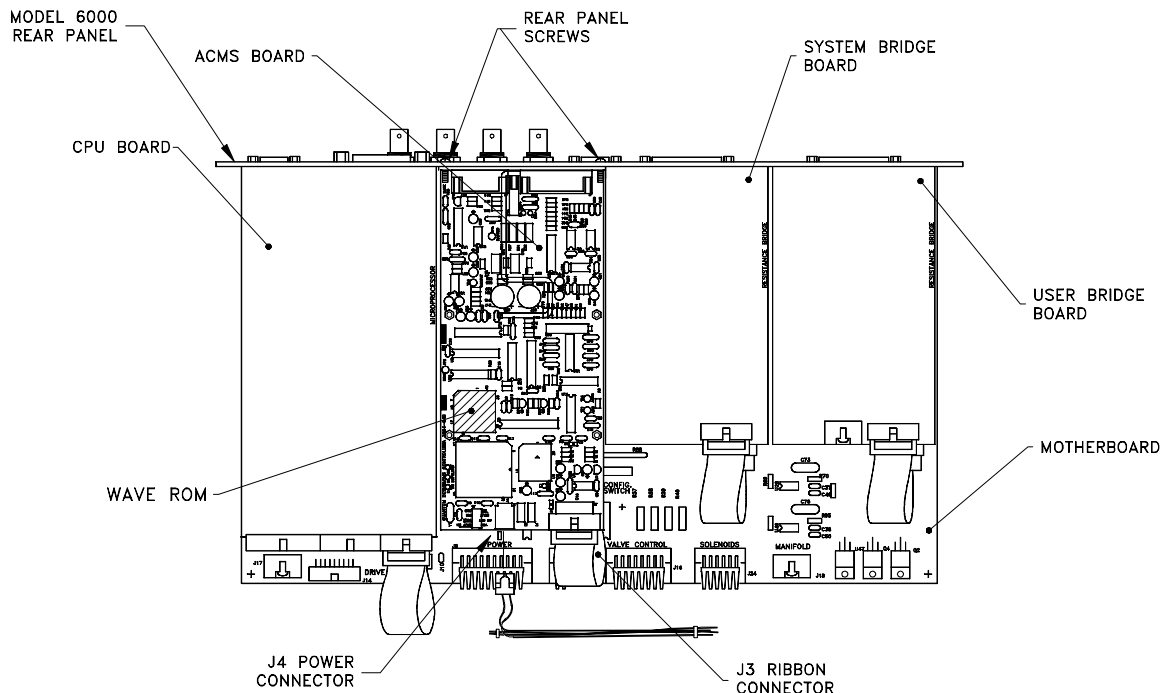


Figure 2-4. WaveROM EPROM on AC Board in Model 6000 PPMS Controller

2.2.4.1 REPLACING THE WAVEROM CHIP

To replace the waveROM chip on the AC board, first locate the upgrade kit, which contains the PLCC chip extraction tool and the new ROM in a small plastic box. Remove the lid of the Model 6000 so that you can access the AC board (note that the AC board may reside in the Model 6500 Option Controller instead). Turn off the Model 6000 and extract the old waveROM by inserting the hooks of the extraction tool in the two slots on opposite corners of the ROM housing and gently squeezing until the chip lifts out. To insert the new ROM, note that the upper left corner (as viewed in Figure 2-4) is notched, and the upper side has the label attached. Press down, applying firm and even pressure to the chip until it is seated in the housing.

2.2.5 Thermal Transport Connection Cable

The Thermal Transport connection cable (part number 3084-582) connects the sample to the Model 7100 AC Transport Controller and to the user bridge board that is in the Model 6000 PPMS Controller. Two separate shielded cables on the connection cable plug into the Model 7100. These separate shielded cables split the sample signal and excitation signal in order to help prevent sample signal distortion by the excitation signal.

Labels on the cable's connectors (Figure 2-5) identify the ports for those connectors. You should also refer to Figure A-1, the "Thermal Transport Option Connection Diagram."

- The 14-pin Lemo connector plugs into the gray, color-coded port on the PPMS probe head.
- The connector labeled "J1 (P1) User Bridge" plugs into the "P1–User Bridge" port on the Model 6000.
- The connector labeled "J2 (P1) Sample Current Out" plugs into the "P1–Sample Current Out" port on the Model 7100.
- The connector labeled "J3 (P5) Sample Voltage In" plugs into the "P5–Sample Voltage In" port on the Model 7100.

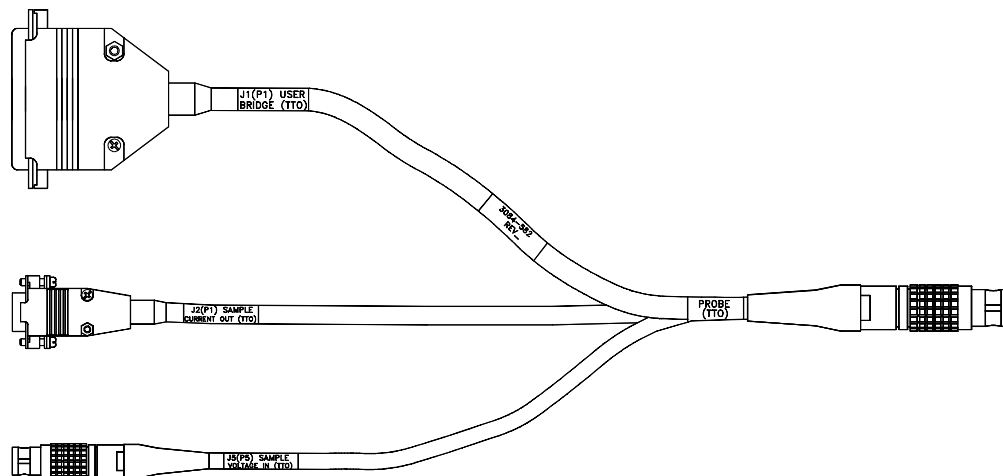


Figure 2-5. Thermal Transport Connection Cable

2.2.6 User Bridge Board

The TTO system employs the user bridge board to read the hot and cold Cernox thermometer shoe assemblies that are on the sample. The user bridge board is in the Model 6000 (see Figure 2-4). Detailed information about the user bridge board is in the *Physical Property Measurement System: Resistivity Option User's Manual*.

2.3 ACT Hardware

The TTO system uses hardware for the AC Transport Measurement System (ACT) option in order to generate the heat pulse and read back the sample thermal voltages in the thermal measurements, and to make the four-probe resistivity measurement on the sample.

2.3.1 Model 7100 AC Transport Controller

The driver board in the Model 7100 AC Transport Controller excites the sample by receiving and amplifying the signal from the AC board's digital signal processor (DSP). The preamp board in the Model 7100 detects the sample signal and sends the signal back to the DSP so the DSP can process the signal. The *Physical Property Measurement System: AC Transport Option User's Manual* discusses the components and operating modes of the Model 7100 in more detail.

CAUTION! The Model 7100 provides as much as 200 mA of current when being controlled by the TTO system. Although this is lower than the hardware limit of 2 A, this current can still damage samples in the current path. Use only currents that can be safely handled by all hardware and samples in the circuit.

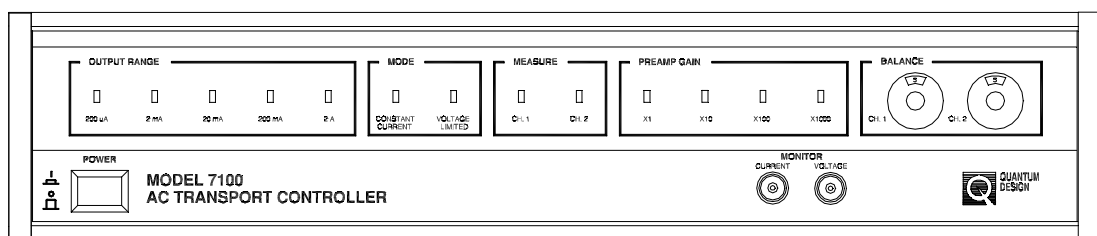


Figure 2-6. Front Panel on Model 7100 AC Transport Controller

2.3.2 AC Board

The AC board is installed in the Model 6000 PPMS Controller and is located behind the “P3–Option” port, which is the port connecting the Model 6000 to the Model 7100. The waveROM EPROM (Section 2.2.4) plugs into the AC board. The AC board includes a DSP, digital-to-analog converter (DAC), current drivers, and other control electronics that are necessary to synthesize excitation signals and process sample response signals. The DSP provides the excitation waveform and processes the sample signal.

2.4 High-Vacuum Hardware

The High-Vacuum option, which operates in conjunction with the TTO system, reduces the amount of gas in the sample chamber and thus minimizes stray thermal conduction from the heated sample. The TTO system works with either the Turbo Pump High-Vacuum option or the Cryopump High-Vacuum option. The details of the Turbo Pump and Cryopump high-vacuum systems are contained in the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* and *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*, respectively.

2.4.1 Contact Baffle

An integral part of either the Turbo Pump or the Cryopump High-Vacuum system is the contact baffle assembly. The contact baffle makes thermal contact with the isothermal region of the sample chamber, which is just above the puck. The thermal contact between the contact baffle and the isothermal region helps create a more uniform thermal environment for the puck by causing the contact baffle to be at the same temperature as the chamber walls that are near the puck. This is important when high vacuum is enabled; high vacuum reduces the amount of thermal exchange gas in the sample chamber.

You insert the contact baffle into the brass fitting that is at the bottom of the baffle assembly (see Figure 2-8B). To help safeguard the contact baffle, use it only when you are using the High-Vacuum option. Handle the contact baffle with care, and avoid touching the delicate outer contact fingers. The charcoal holder is used to achieve the best vacuum at low temperatures and should always be screwed onto the bottom of the contact baffle when in the high-vacuum state. However, when performing a temperature calibration of any sample hardware, such as TTO shoe assemblies, the charcoal holder should be removed to ensure that adequate thermal exchange gas remains in the sample chamber at low temperature.

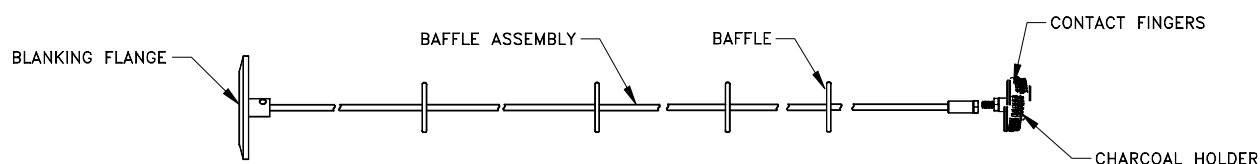


Figure 2-7. Baffle Assembly with Contact Baffle

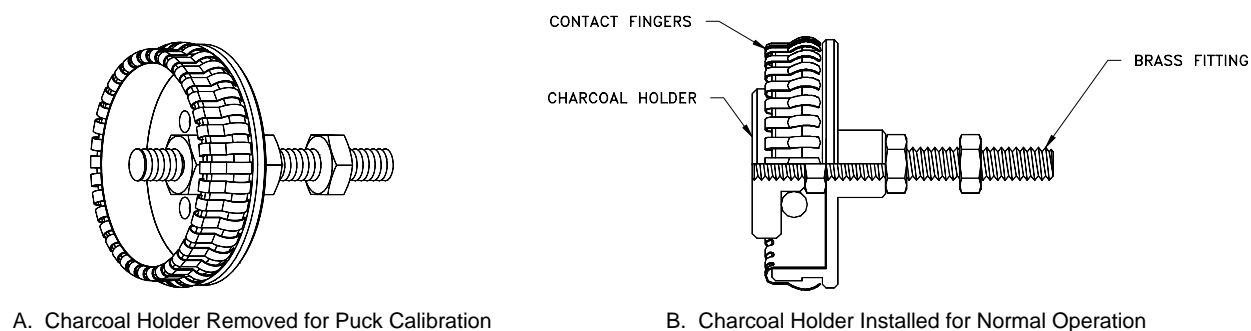


Figure 2-8. Close-up View of Contact Fingers and Charcoal Holder on Contact Baffle Assembly

2.5 Calibrating New Shoe Assemblies

A spare set of uncalibrated shoe assemblies (two thermometers and one heater) is included in the TTO user's kit. You can easily calibrate the shoe assemblies by using the calibration wizard (see Figure 2-10) that is accessed in the **Advanced** tab of the Thermal Transport control center. To calibrate new shoe assemblies, the calibration fixture (3084-576) must first be plugged into the Thermal Transport puck as shown in Figure 2-9.



The vertical plate that is usually mounted between the sample and the plugs for the shoe assemblies must be removed before plugging in the calibration fixture. Unscrew the two Phillips-head screws at the base of the plate only enough to remove the plate, and then retighten the screws to hold the PC board. Use caution so that you do not strain the wiring on the bottom side of the PC board: do not lift or turn the board or pinch any wires when retightening the screws.

If a heater shoe is being calibrated, plug it into the left-hand socket (the socket closest to the marking "PCB 3084-576" –see Figure 2-9). If calibrating thermometer shoe assemblies, plug them into the other two sockets, with "Thermometer A" in the middle socket and "Thermometer B" in the right-hand socket. Note that wiring in the shoe assemblies is symmetric, so plugging in the connectors in either of the two possible orientations will make the proper electrical connections.

Next, locate a gold-plated copper calibration sample from the TTO User's Kit. Bend the leads on the sample so that you can mount it as shown in Figure 2-9. Then mount the copper sample to the cold foot and mount the copper shoes to the sample. When mounting the heater shoe, make sure the copper sample does not touch the solder pad of the heater resistor, which could cause an electrical short.

Note the serial numbers of each shoe assembly. Then screw the shield onto the TTO puck. Unscrew the shield cap and make sure none of the copper shoes are touching any part of the puck or the shield. Replace the shield cap; then insert the puck into the PPMS. Remove the charcoal carrier on the contact baffle assembly (see Section 2.4.1) to ensure exchange gas is not cryopumped at low temperatures. Place the baffle set inside the sample chamber. Then purge and seal the sample chamber.

In the calibration wizard window, check the box for the thermometers and/or heaters you wish to calibrate and enter their serial numbers. The default temperature range should be 1.8 to 400 K. The heater parameters have been selected to optimize the signal for the 2k Ω heater resistors supplied. Press the "Start" button to begin calibration, which will last approximately 16 hours.

After calibration is complete, you must make the appropriate changes to TTO initialization file TTO.INI if you wish to use the newly calibrated shoe assemblies immediately. See Section 3.2.

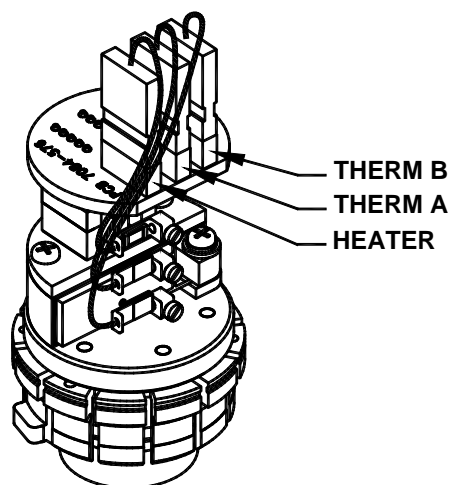


Figure 2-9. Calibration Fixture Plugged into TTO Puck and Illustrating Sockets for Each Shoe Assembly

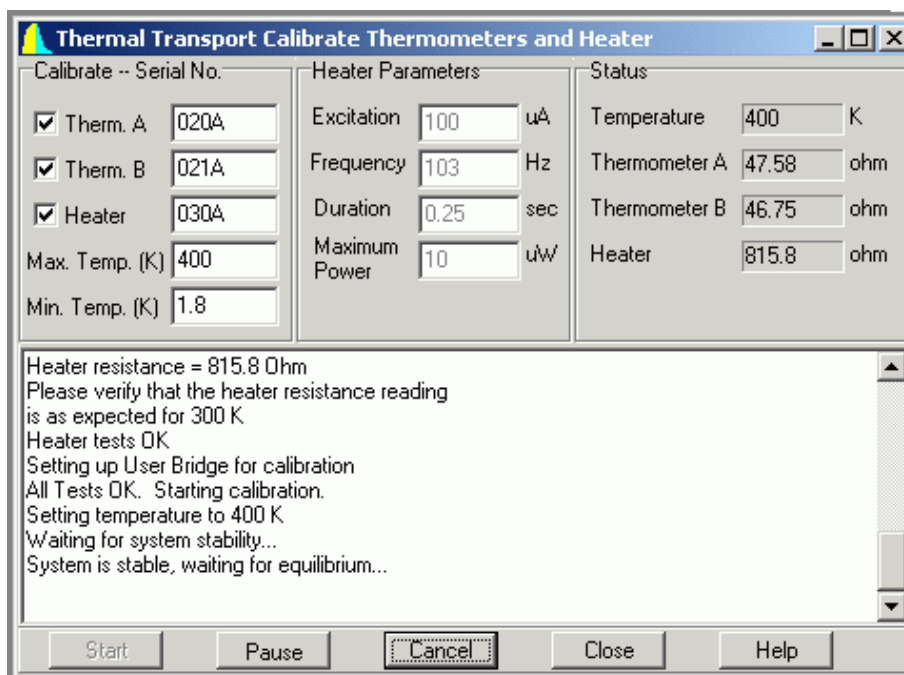


Figure 2-10. Thermal Transport Calibrate Thermometers and Heater Wizard

Software

3.1 Introduction

This chapter contains the following information:

- Section 3.2 presents an overview of the TTO system software.
- Section 3.3 discusses the Thermal Transport control center.
- Section 3.4 discusses the TTO data files.
- Section 3.5 explains how to examine data saved to a TTO data file.

3.2 Overview of Thermal Transport Software

The TTO software module is integrated into the Quantum Design PPMS MultiVu environment. Version 1.1.6 or greater of the PPMS MultiVu software is required to install the TTO software. For software installation instructions, see Appendix A.

TTO is designed to be used by both experts and newcomers to the study of thermal transport properties. To accommodate this spectrum of users, the software interface is multi-leveled so that beginners can easily set up and perform measurements at the top level while experts can choose to navigate into submenus in order to adjust various parameters or customize measurements.

TTO measurements can be run either interactively at the dialog or in a sequence program. While the TTO software is active in PPMS MultiVu, the command **TTO Measure** appears in the sequence command bar under the **Measurement Commands** heading. This command is used to start, stop, or change measurement parameters within a sequence file. Users who are familiar with measurement sequence commands for other Quantum Design options will find an important difference in the use of the **TTO Measure** command compared to other options, where sequence execution is typically paused until a measurement command is completed. This principle is not appropriate for continuous TTO measurements because these can be performed while a system parameter such as the temperature is swept slowly. Therefore, the **TTO Continuous Measure** command initiates measurements and the sequence execution immediately continues. TTO measurements are taken until the **TTO Stop** sequence command is issued. The TTO single measurement sequence commands are more similar to the traditional measurement commands. During these measurements, sequence execution is paused until the measurement is completed because single measurements operate on the condition that system parameters are stable during the measurement.

The data file for TTO measurements (*sample.dat*, where *sample* is the file name you select) contains the results for each measurement that was completed successfully. The raw data from all measurements can also be saved in a separate file (*sample.raw*).

As soon as the TTO software is activated in PPMS MultiVu, the **Thermal Transport Log** window (Figure 3-1) opens and indicates which thermometer and heater calibration files will be used. These settings, as well as several other parameters, can be edited in the *Tto.ini* initialization file located in the QdPpms\ThermalTransport\System directory. The thermometer and heater calibration files are located in the QdPpms\ThermalTransport\Calibration directory. The TTO log can be viewed by using the **View>TTO Status Log** menu command.

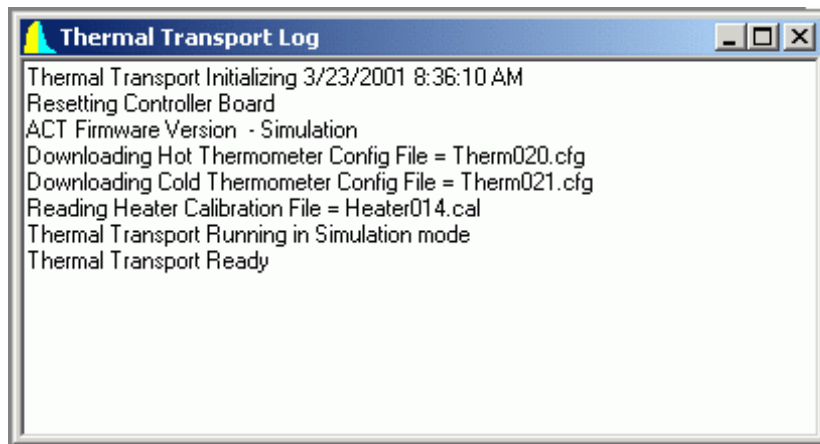


Figure 3-1. **Thermal Transport Log** Window

3.2.1 Measurement Units

Thermal conductivity κ is typically stated in either units of watt/(meter-kelvin), written W/m-K, or in mW/cm-K. In the TTO software, the units of W/m-K are used for thermal conductivity. The Seebeck coefficient is expressed most conveniently in $\mu\text{V/K}$, because in many electrical conductors $\alpha \sim \mu\text{V/K}$ at room temperature. The electrical resistivity ρ is stated in units of ohm-meter ($\Omega\text{-m}$). The thermoelectric figure of merit $ZT = \alpha^2 T / \rho \kappa$ is dimensionless when MKS units are chosen for each quantity; that is, $\alpha[\text{V/K}]$, $T[\text{K}]$, $\rho[\Omega\text{-m}]$, and $\kappa[\text{W/m-K}]$.

3.3 Thermal Transport Control Center

The TTO software module has a control center that includes all frequently selected Thermal Transport commands. With its easy-to-use tab format and software prompts, the control center makes basic system operations, such as installing samples, creating data files, and setting up and running immediate-mode measurements, more natural and convenient. The Thermal Transport control center opens as soon as the Thermal Transport option is activated in PPMS MultiVu, and although it may be minimized, does not close until the option is deactivated. Figures 3-2 through 3-6 illustrate the tabs in the Thermal Transport control center.

3.3.1 Control Center Tabs

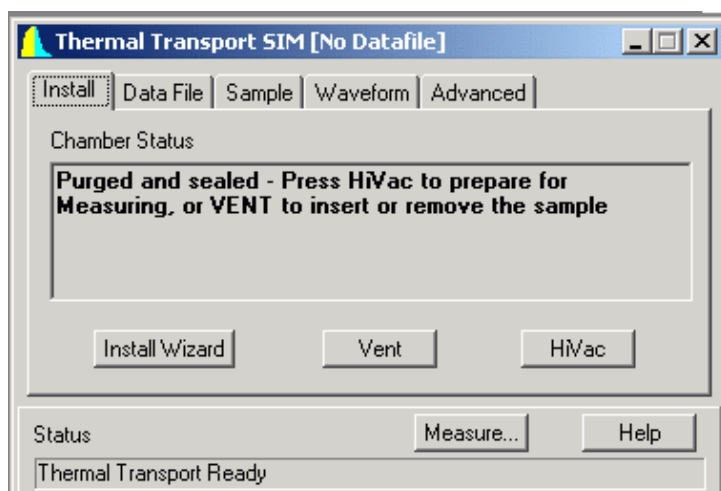


Figure 3-2. Control Center **Install** Tab

The **Install** tab automatically opens when the software starts, and it assists you in sample installation or removal by providing sample chamber **Vent** and **HiVac** buttons and by also providing an install wizard with more extensive, step-by-step instructions for setting up a measurement.

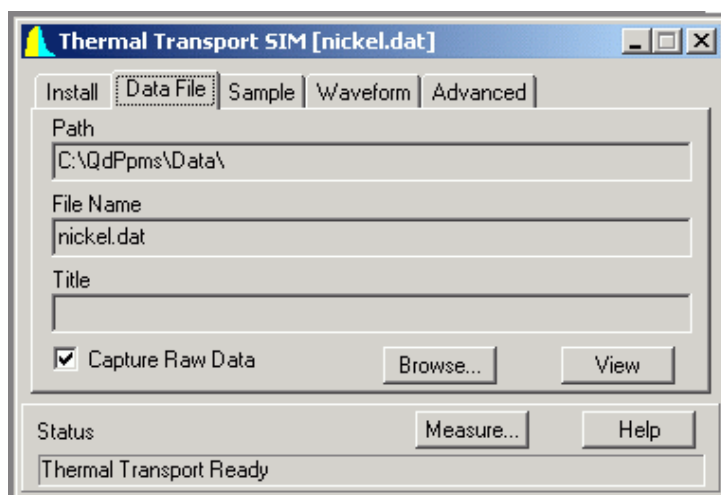


Figure 3-3. Control Center **Data File** Tab

The **Data File** tab lets you open a new TTO data file or append to an existing TTO data file. You can also use the tab to view data and select whether raw thermal transport data is saved in a .raw file. See Section 3.4 for information on TTO data file formats.

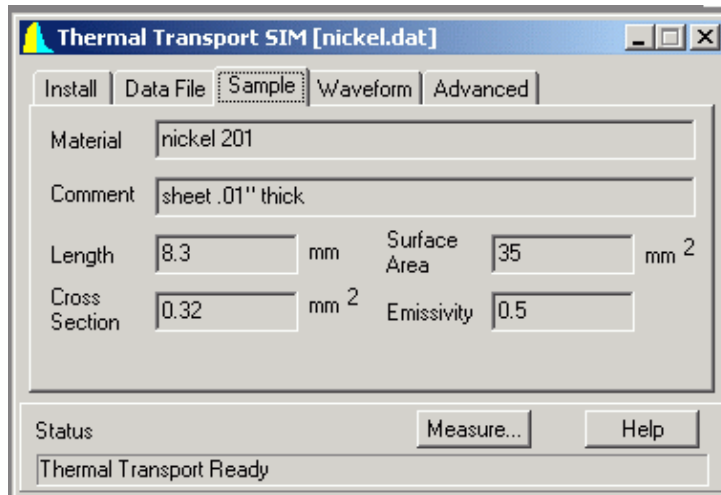


Figure 3-4. Control Center **Sample** Tab

The **Sample** tab displays the sample name, comments, geometry and radiation estimates for the currently open data file. These parameters can be changed only by opening a new file in the **Data File** tab. Chapter 4 has more information on how to mount sample leads and estimate properties such as the sample's infrared emissivity.

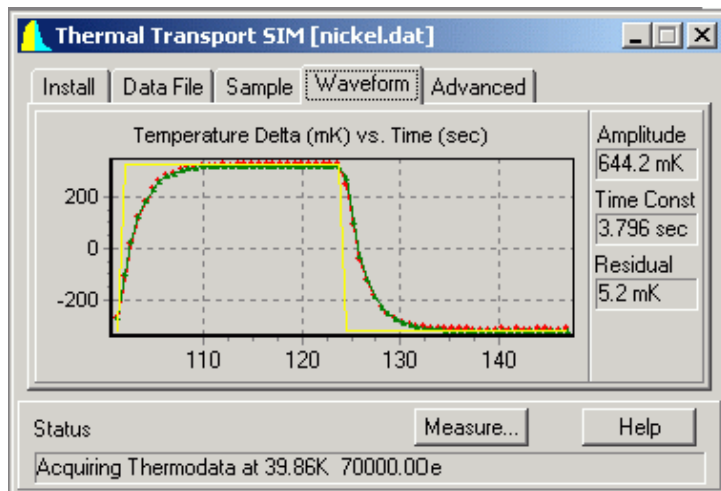


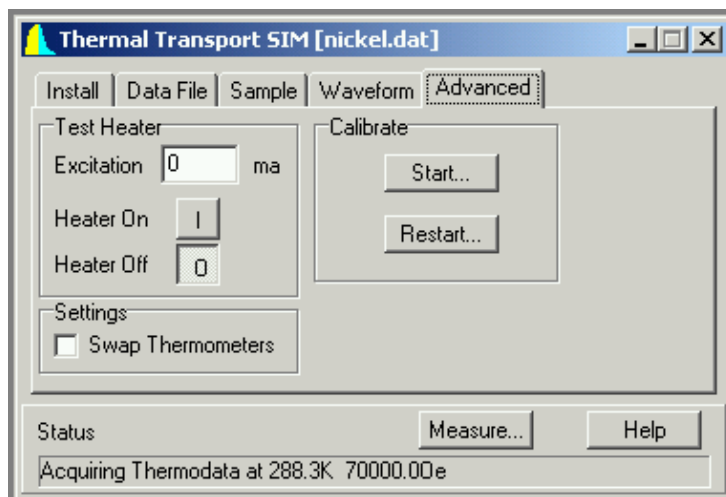
Figure 3-5. Control Center **Waveform** Tab

The time traces of the most recent raw data, along with the fitted curves, are displayed in the **Waveform** tab. As a visual aid, the heater pulse is shown schematically in the **Waveform** tab as a yellow square wave in both the thermal conductivity and Seebeck graphs.

Clicking the right mouse button inside the graph window in the **Waveform** tab opens a menu that allows selection of thermal conductivity, Seebeck coefficient, or resistivity results, and whether to plot the fitted curve along with raw data. The title of the graph indicates which data is being displayed. For thermal conductivity data, "Temperature Delta" refers to the difference between hot and cold thermometers, while for Seebeck measurements "Seebeck Voltage" refers to the voltage difference between the hot and cold shoes.

To the right of the graph are listed three parameters that briefly summarize the curve fitting results: the total amplitude obtained by the curve-fitting routine (equivalent to Delta Temp. or Seebeck Volt. in the data file), the long time constant in the measurement (that is, τ_1 —this is not relevant to resistivity data), and the residual of the curve fit (that is, the error estimate for the reported total amplitude). Section 1.5 contains more information on the AC measurements and error estimation in TTO data.

In the **Waveform** tab, you can also zoom to examine details of the data by dragging the mouse from the upper left to the lower right corner of the graph while holding down the left mouse button. To zoom out, drag the mouse in the opposite direction while holding down the button, or select **Zoom All** in the pull-down menu, which is accessed by clicking the right mouse button in the graph.

Figure 3-6. Control Center **Advanced** Tab

The **Advanced** tab includes a software wizard that allows you to calibrate new TTO shoe assemblies (heater and/or thermometers) in the PPMS. The calibration fixture (3084-576) from the user's kit is used in conjunction with this software wizard. The **Restart...** button is used if you previously cancelled a calibration and want to restart. Refer to Section 2.5 for more information on calibrating shoe assemblies.

Other features on the **Advanced** tab include a heater test in which you select the desired heater current to apply, as well as an option to swap the software's assignment of hot and cold thermometers.

3.3.2 Measurement Menu

Selecting the **Measure...** button at the bottom of the Thermal Transport control center opens the **Thermal Transport Measurement** dialog box, which allows you to run immediate-mode TTO measurements without having to write a sequence file. Three tabs—**Settings**, **Thermal**, and **Resistivity**, shown in Figures 3-7, 3-8, and 3-9—are immediately visible in the **Thermal Transport Measurement** dialog.

In the **Settings** tab (Figure 3-7) you determine basic settings for all measurements. First you select which of the four thermal transport quantities are to be measured. Note that checking **Figure of Merit (ZT)** automatically selects all measurements because they are all required to assess ZT. You have the option to save "marginal" results, which would otherwise be discarded, to the data file. Marginal measurements are defined as those for which the software was able to determine a quantity, but the regression errors of the curve fits were between 50% and 200% (the measurement is considered failed if the regression is higher than 200%). The **Discard First N Results** check box is included because the first several measurements often have large errors due to the measurement parameters not being optimized. Choosing $N = 3$ is typically adequate, if you choose to discard any results. In the **Next Measurement** box you can specify the period and heater power for the next thermal measurement cycle, even after measurements have started. Note that any changes made in this box are saved only if the **Set** button is selected. Selecting **Clear** restores the values that were last input.

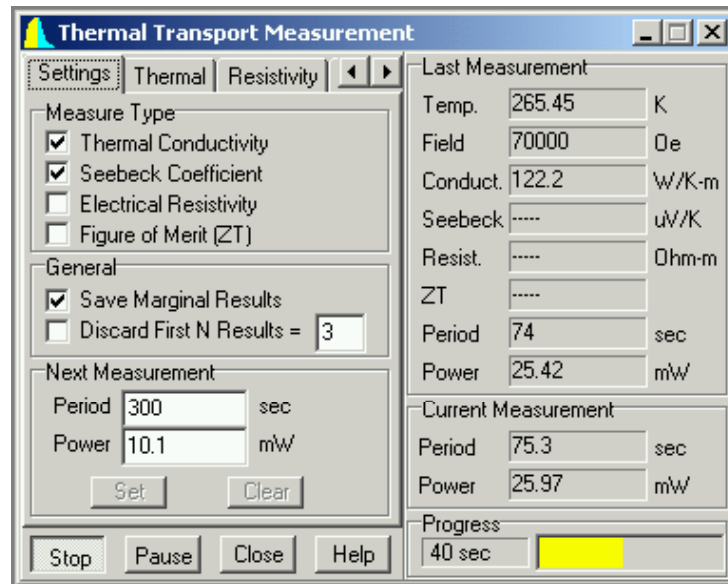


Figure 3-7. **Settings** Tab in **Thermal Transport Measurement** Dialog Box

Settings and limits for thermal measurements (thermal conductivity and Seebeck coefficient) are determined in the **Thermal** tab (Figure 3-8). These settings and limits include limits of heater period and power and expected limits of Seebeck readback voltage, as well as target amplitude for the heat pulse (expressed as percentage of sample temperature) and target value of period ratio, which is defined as measurement period divided by the time constant (τ_{av}) of the sample (see Section 3.4.3 for more information on these quantities). Any changes made in this tab are saved only if the **Set** button in the tab is selected. A ToolTip displays hardware limits for the heater power if the cursor is placed over the panels showing the heater power limits. Note that a period ratio of at least 8 is recommended because choosing a value lower than this has been shown to produce artifacts in the thermal transport measurements due to insufficient data for the software model.

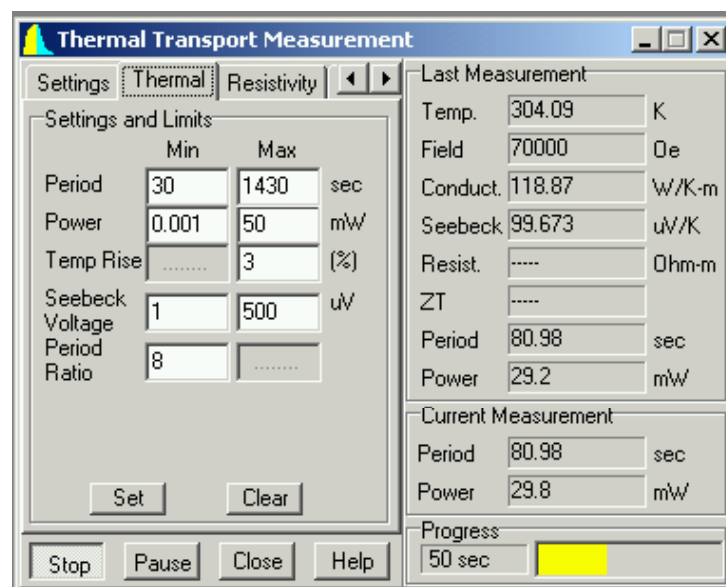


Figure 3-8. **Thermal** Tab in **Thermal Transport Measurement** Dialog Box

Due to the complexity of making an adaptive resistivity measurement, a separate tab is devoted to resistivity (Figure 3-9) so that you can have maximum freedom in setting limits such as min/max excitation and min/max frequency, as well as measurement duration. The measurement excitation frequency is allowed to vary so that a 90% resistive (that is, in-phase) signal is obtained. Autoranging parameters may also be changed, with the default being **Sticky Autorange**. Changes made in this tab are saved only if the **Set** button in the tab is selected. See the *Physical Property Measurement System: AC Transport Option User's Manual* for more information on resistivity measurement parameters.

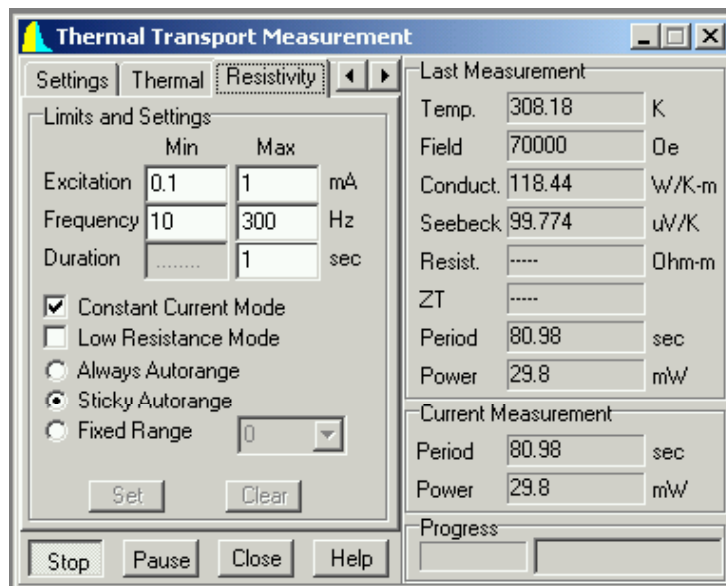


Figure 3-9. **Resistivity** Tab in **Thermal Transport Measurement** Dialog Box

3.3.2.1 OPTIONS FOR ADVANCED USERS

Two more tabs, the **Mode** tab and the **Advanced** tab, are made visible in the **Thermal Transport Measurement** dialog box by clicking on the right-pointing arrow that is next to the tab names at the top of the dialog box. Clicking on the left-pointing arrow hides the **Mode** and the **Advanced** tabs. Generally, these two tabs are of interest only to advanced users.

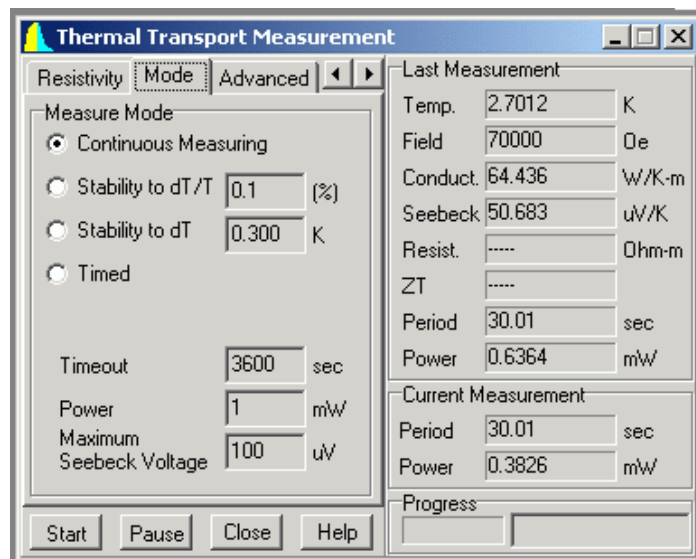


Figure 3-10. **Mode** Tab in **Thermal Transport Measurement** Dialog Box

You use the **Mode** tab to select whether to take continuous measurements (default) or single, steady-state measurements. There are two modes of single measurement: stability or timed. Section 1.3 describes the measurement modes in more detail.

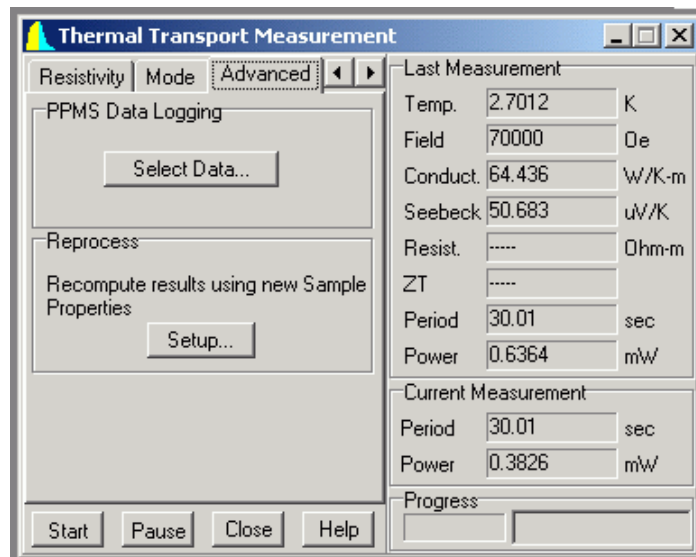


Figure 3-11. **Advanced** Tab in **Thermal Transport Measurement** Dialog Box

The PPMS data logging dialog can be accessed from the **Advanced** tab in the event that you would like to select which PPMS status items are written to the TTO data file. Table 3-1 on the following page describes the parameters that can be monitored on the PPMS data logging dialog. The **Reprocess** command allows you to recompute thermal transport results from an existing TTO data file (*.dat) by changing the sample's estimated geometry and emissivity, and the command allows you to write these results out to a new data file.

Table 3-1. PPMS system data items that can be saved to the TTO measurement data file. Items in bold and all capital letters are always written to the data file.

ITEM	DEFINTION
GENERAL STATUS	General system status, coded. Appendix B explains how to interpret status code.
TEMPERATURE	System temperature, measured at base of sample chamber.
MAGNETIC FIELD	Magnetic field.
Sample Position	For use with rotator probes*.
SAMPLE PRESSURE	Pressure in sample chamber, measured in torr.
Digital Inputs	Eight-bit status of selected inputs.
Dr Ch 1–2 Current	Current delivered by selected driver output channel.
Dr Ch 1–2 Power	Power delivered by selected driver output channel.
Brg Ch 1–4 Resistance	Resistance of selected user bridge channel.
Brg Ch 1–4 Excitation	Excitation current of selected user bridge channel.
Sig Ch 1–2 Input Voltage	Input voltage for selected signal channel.
Map 20–29 MAP 21–22	User-designated data items. Reserved for hot and cold sample thermometers.

* Rotator probes are not currently available for use with TTO.

3.3.3 System Status

Features in the Thermal Transport control center provide constant updates of TTO status.

- The **Status** bar at the bottom of the Thermal Transport control center succinctly describes the progress of an on-going measurement and summarizes the results of the last measurement. Color-coded warning and error messages in the **Status** bar alert you to possible problems. Warning messages appear on a yellow background. Error messages appear on a red background.
- The **Waveform** tab in the Thermal Transport control center (Figure 3-5) displays raw data waveforms and curve fits for any of the measurements, and the tab indicates the fitting parameters of signal amplitude, time constant τ_1 (where relevant) and the residual for the fit.
- The measurement **Progress** bar and countdown timer in the bottom right corner of the **Thermal Transport Measurement** dialog box indicate the time remaining before the measurement is complete. Refer to Figure 3-7. In the **Progress** bar, yellow indicates the heater “on” state while blue indicates “off.”
- The right side of the **Thermal Transport Measurement** dialog box summarizes the most recent measurement and the parameters (period and power) used for the current measurement. Refer to Figure 3-8. Note that if the cursor is placed over the displayed results, a ToolTip shows the estimated error for the results.
- The **Thermal Transport Log** window (Figure 3-1) keeps a record of all messages that appear in the Thermal Transport control center **Status** bar. The TTO status log is saved in the file `TtoLog.txt`, which is in the directory `C:\QdPpms\ThermalTransport\LogFiles`.

3.4 Thermal Transport Data Files

Thermal Transport measurement data is stored in measurement data files and raw measurement data files. Measurement data files, which have a `.dat` extension, store relevant sample measurement data and selected system data for any number of measurements taken by any of the Thermal Transport measurement types. Raw measurement data files, which have a `.raw` extension, store raw temperature versus time (thermal conductivity) and voltage versus time (Seebeck and resistivity) data as well as the software's fits to the data. Raw data is saved only if the **Capture Raw Data** check box in the **Data File** tab (Figure 3-3) is selected. The system data items that may be recorded are user configurable (Section 3.4.2).

The results from measurements, whether made in continuous or single mode, are automatically saved if a data file is open. Measurement and system data is appended to the selected data files and is never overwritten. The **Data File** tab in the Thermal Transport control center identifies the selected measurement data file and raw measurement data file and includes a **Browse** command button that enables data file selection and creation. The name of the selected measurement data file is also displayed in the title bar of the Thermal Transport control center.

3.4.1 Saving Raw Thermal Transport Data

Raw thermal transport data is saved to a separate raw measurement data file that has the identical base name as the measurement data file, but a `.raw` file extension instead of a `.dat` extension. Saving the raw data can be useful when you are deciding which measurement parameters to use or if you are concerned about signal quality. A clean, single-wavelength sine wave is optimal in the case of AC resistivity measurements. In the cases of thermal conductivity and Seebeck measurements, the fitted model curve can be plotted along with the raw data so that you can easily assess the quality of the results. However, saving raw data creates very large data files.

Raw thermal transport data is saved only if a measurement data file is selected *and* the software is prompted to capture raw data. Enabling the **Capture Raw Data** check box in the Thermal Transport control center's **Data File** tab (Figure 3-3) prompts the software to save raw data. By default, this check box is *not* selected.

3.4.2 Data File Header

The data file header contains file and sample property information that is defined when the data file is created. Information written to the data file header cannot be subsequently changed in PPMS MultiVu. The file information that can be written to the header consists of the title assigned to the graph view of the data file. The sample property information consists of the sample dimensions and emissivity. User comments can also be written to the header. All this information appears in the INFO declarations of the header. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* contains more information on data file headers.

When you create a data file, the software prompts you to enter the physical dimensions and an estimate of the emissivity for the sample whose measurement data will be saved to the file. To enable you to do this, the data entry fields in the Thermal Transport control center's **Sample** tab are enabled. As soon as you define the sample properties and select **OK**, all data entry fields in the **Sample** tab (Figure 3-4) are disabled again.



Because the data file header identifies a particular sample, changing samples without changing data files can destroy the validity of the data in the file. Therefore, you are encouraged to use the automated routines in the Thermal Transport control center. These automated routines prompt you for new data file(s) and new sample information after you install a different sample.

3.4.3 Format of Measurement Data Files

Table 3-2 lists and describes all data columns in the Thermal Transport data file. Section 5.3 provides more information on some of these fields.

Table 3-2. Fields in Thermal Transport Measurement Data File

ITEM	DEFINITION
Time Stamp	Time of measurement data point, expressed in minutes or seconds, and as an absolute time or relative to the start time of the data file.
Status (code)	PPMS system status. Identical to General Status in Table 3-1.
Error (code)	TTO error code. Appendix B describes how to interpret the code.
Magnetic Field (Oe)	Magnetic field.
Sample Temp. (K)	Average sample temperature during measurement.
Conductivity (W/K-m)	Sample thermal conductivity.
Cond. Std. Dev.	Error (standard deviation) in thermal conductivity measurement.
Seebeck Coef. (uV/K)	Sample Seebeck coefficient in units of $\mu\text{V/K}$.
Seebeck Std. Dev.	Error in Seebeck coefficient measurement.
Resistivity (Ohm-m)	Sample resistivity.
Resist. Std. Dev.	Error in resistivity measurement.
Figure of Merit [ZT]	Dimensionless thermoelectric figure of merit ZT.
Merit Std. Dev.	Error in ZT measurement.
Delta Temp. (K)	Extrapolated (asymptotic) temperature drop ΔT across heated sample.
Conductance (W/K)	Net thermal conductance of sample. See Section 1.5.5.
Raw Conductance (W/K)	Raw thermal conductance, that is, (Heater Power)/(Delta Temp.).
Seebeck Volt. (uV)	Extrapolated (asymptotic) Seebeck ΔV across heated sample.
Resistance (Ohm)	Sample resistance.

(continues)

Table 3-2. Fields in Thermal Transport Measurement Data File (Continued)

ITEM	DEFINITION
Min. Temp. (K)	Minimum temperature at either hot or cold thermometer during measurement.
Max. Temp. (K)	Maximum temperature at either hot or cold thermometer during measurement.
Temp. Rise (K)	Rise in temperature of the hot thermometer due to the applied heat pulse. Should be close to user-requested value set in Thermal tab.
Req. Htr. Power (W)	Requested heater power, in watts.
Heater Power (W)	Actual heater power.
Rad. Loss (W)	Estimated power loss due only to radiation from sample. See Section 1.5.5.
Cond. Pwr. (W)	Estimated net power conducted through sample.
Heater Current (mA)	Current through heater.
Res. Drive (mA)	Current drive used for resistivity measurement.
Res. Freq. (Hz)	Frequency used for resistivity measurement.
Period (sec.)	Period for heater on/off square-wave pulse.
Period Ratio	Ratio of period/tau1.
tau1 (sec.)	Long thermal time constant of sample and shoes.
tau2 (sec.)	Short thermal time constant of sample and shoes.
Seebeck Gain	Total gain (preamplifier and DSP) for Seebeck data point.
Resist. Gain	Total gain (preamplifier and DSP) for resistivity.
System Temp. (K)	PPMS block system temperature.
Sample Position (deg.)	Used with rotator probes; not used in TTO*.
Brg Ch 1–4 Resistance	Resistance of selected user bridge channel.
Brg Ch 1–4 Excitation	Excitation current of selected user bridge channel.
Sig Ch 1–2 Input Voltage	Input voltage for selected signal channel.
Digital Inputs (code)	Eight-bit status of selected inputs.
Dr Ch 1–2 Current	Current delivered by selected driver output channel.
Dr Ch 1–2 Power	Power delivered by selected driver output channel.
Pressure	Sample chamber pressure, in torr.
Map 20–29 Map 21–22	User-designated data items. Reserved for hot and cold sample thermometers.

* Rotator probes are not currently available for use with TTO.

3.4.4 Format of Raw Data Files

If the **Capture Raw Data** check box is selected in the **Data File** tab in the Thermal Transport control center (Figure 3-3), raw measurement data is recorded to a data file. For thermal conductivity and Seebeck coefficient measurements, raw data also includes results from the thermal model. For resistivity measurements, raw data includes the excitation current and signal voltage.

Table 3-3. Fields in Thermal Transport Raw File

ITEM	DEFINITION
Comment	System status and TTO software comments.
Time Stamp	Time of measurement data point, expressed in minutes or seconds, and as an absolute time or relative to the start time of the data file.
T-Hot (K)	Temperature of sample hot thermometer.
T-Cold (K)	Temperature of sample cold thermometer.
T-Sys (K)	Temperature of PPMS system thermometer.
Delta T (K)	Temperature drop across sample thermometers.
Model Delta T (K)	Curve fit of ΔT to software thermal model.
Seebeck (μV)	Raw Seebeck voltage.
Model Seebeck (μV)	Curve fit of Seebeck to software thermal model.
Res. Excit. (mA)	Excitation current from resistivity measurement.
Res. Signal (mV)	Signal voltage from resistivity measurement.

3.5 Data Examination

To examine the current TTO .dat data file in PPMS MultiVu, you can select the **View** button in the **Data File** tab in the control center. If raw data is also being saved, the raw file can be opened by using the **File>Open** menu command. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses the graphing and data-viewing formats in detail.

The ExportData.exe program, located in the QDPPMS\Tools directory, can be used to export specific columns and portions of the data file header from any data file.

Errors encountered by the PPMS during TTO data acquisition are listed in the TTO status log (see Section 3.3.3). In addition, an **Error Count** dialog is opened if any individual TTO measurement fails. A running total of the error counts is displayed and brief one-line descriptions of the last three errors are listed in the window. If this dialog is closed, it can be accessed by using the **View>TTO Error Count** menu command. Selecting the **Reset** button will zero the displayed error totals for each measurement, but will not affect the TTO log file.

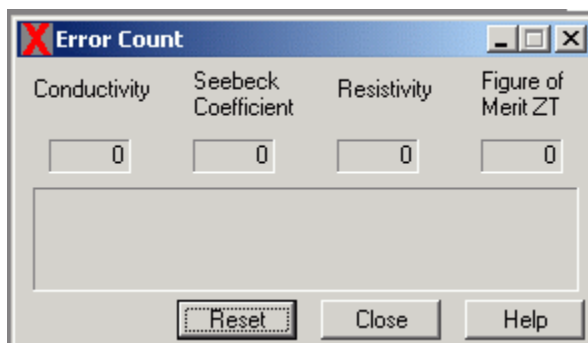


Figure 3-12. **Error Count** Dialog Box

Sample Preparation

4.1 Introduction

This chapter contains the following information:

- Section 4.2 discusses sample-mounting considerations.
- Section 4.3 discusses the two-probe and four-probe lead configurations.
- Section 4.3 explains how to check the sample contact.
- Section 4.5 explains how to use the puck-mounting station.

4.2 Sample-Mounting Considerations

Four leads must be attached to the sample in order for the TTO system to take thermal (and electrical) measurements. These four leads are a heater (and current I+), a heat sink (and current I-), and two temperature (and voltage) probes that are along the length of the sample. The TTO system takes both thermal and electrical measurements by using the same probes, so measurements of thermal conductivity, thermopower, and electrical resistivity can be performed in one pass without remounting the sample.

It is important that the resistance—either thermal or electrical—at the interface between the leads and the sample be minimized. This is especially important when a two-probe measurement (Section 4.3.1) is performed, because any contact resistance is directly reflected in the measured sample thermal and electrical resistance. In addition, you are advised to minimize the thermal diffusion time in the leads by keeping them short (2–3 millimeters, if possible), because this expedites the measurement process.

4.2.1 Geometry

The geometry of the sample is constrained due to a variety of considerations, the most obvious of which is the Thermal Transport sample puck. Mounted vertically on the puck, a sample cannot be much longer than 20 mm, while the minimum convenient sample length is typically 3 mm.

Another aspect to consider is the thermal diffusion time in the sample, defined as $\tau \sim C/K$, where C (Joule/K) is the heat capacity and K (Watt/m) is the thermal *conductance* of the sample. This places an operational lower limit on K so that the measurement time does not become excessively long (one measurement is typically designed to be $\sim 8 \times \tau$; see Section 1.5). Since $K = \kappa \times A/l$, where κ (W/m - K) is the thermal *conductivity*, A is the cross-sectional area of the sample, and l is the length, this implies a lower limit on A/l for a given value of κ . Another relation that can be easily derived from the above equations is $\tau \sim c_p \times l^2/\kappa$, where c_p (J/m³ - K) is the specific heat of the material. This implies that, for a given material, the time constant simply scales quadratically with the length, with a typical practical upper bound of $l_{max} \sim 10$ mm on samples.

While the thermal diffusion time τ places a lower limit on A/l , the heater power $P(W) = \kappa \times \Delta T \times (A/l)$, where $\Delta T \sim 0.03 \times T$ (typical value) is the temperature drop across the sample, sets the upper limit on A/l because the heater is limited by the 10-V compliance limit of the Model 7100 current source. For an $R = 2$ - k Ω chip heater, $P_{max} = V^2/R = 50$ mW.

Keep in mind that the constraints mentioned here are most stringent at high T , where τ is generally longer and the required ΔT is larger. Table 4-1 gives some examples of sample geometries and the range of measurable thermal conductivities based on the above considerations and using a 2-k Ω heater.

Table 4-1. Sample Geometries and Range of Measurable Thermal Conductivities

SAMPLE	DIMENSIONS $L \times (A)$ (mm ³)	HIGH T CONSTRAINTS ON κ (W/m - K)
Brick	$8 \times (2 \times 2)$	2–30
Needle	$10 \times (1 \times 1)$	10–150
Pill	$3 \times (5 \times 5)$	0.1–1.5

4.2.2 Lead-Mounting Epoxies

After deciding on an appropriate geometry for the particular thermal transport measurement you will take (see Section 4.2.1), you cut and sand the sample so that its surfaces are clean.

The epoxies used to attach leads to samples should be chosen for the following:

- Strength in bonding to the particular sample material
- High thermal conductivity
- Convenience of the curing schedule

In addition, any time the thermoelectric or electrical properties will be measured, an electrically conducting silver-filled epoxy must be used. On the other hand, the primary advantage of electrically nonconductive epoxies is their very high strength.



Use a generous (but not excessive) amount of epoxy when attaching leads so that the bond is strong and provides very good thermal contact.

The sample-mounting kit contains starter samples of epoxies, including Silver-Filled H20E from Epoxy Technology, Inc., and nonconductive Tra-Bond 816H01 from Tra-Con, Inc. Product specifications and Materials Safety Data Sheets (MSDS's) for both epoxies are included in the epoxy kit.

4.2.2.1 SILVER-FILLED H20E EPOXY

Small amounts of parts A and B (labeled on the containers) should be thoroughly mixed in an approximately 1:1 ratio on a clean, dry, and nonabsorbing surface, using care that no cross-contamination of the remaining portions in the jars occurs. After applying the mixture to the leads and sample and attaching the leads, you can bake the sample at $\sim 150^{\circ}\text{C}$ for about 5 minutes. See the vendor's product data sheet included in the epoxy kit for more information, including other possible curing schedules.

4.2.2.2 TRA-BOND 816H01 EPOXY

This electrically nonconductive epoxy is provided in one-use, 2-gram packets. Because 2 g is a much larger amount of epoxy than is needed for one sample, Quantum Design recommends preparing several samples at the same time. To mix, slide off the plastic clamp and knead both chambers thoroughly so that the texture feels uniform; this occurs after approximately 2 minutes of mixing. Cut open one end of the packet to dispense epoxy, and allow 24 hours for epoxy bonds to cure. Note that the liquid epoxy may have a grainy appearance; this is normal.

4.3 Two-Probe and Four-Probe Lead Configurations

There are two methods of mounting the electrical/thermal leads on a sample. These two different methods present a trade-off between convenience of mounting and accuracy of measurement.

4.3.1 Two-Probe Lead Configuration

The two-probe lead configuration method is the most convenient because it involves attaching only two leads, but this method sacrifices accuracy because heater/I+ and $T_{\text{hot}}/V+$ share one lead while coldfoot/I- and $T_{\text{cold}}/V-$ share the other lead (Figure 4-1). Thus, the thermal and electrical contact resistances between the leads and sample contribute to the measured quantities. You should use the two-probe lead configuration method only when the thermal and electrical resistances of the sample are far greater than those of the leads. Examples of samples mounted in this fashion are shown in Figure 4-1, where both bar-shaped and disk-shaped copper leads are used.

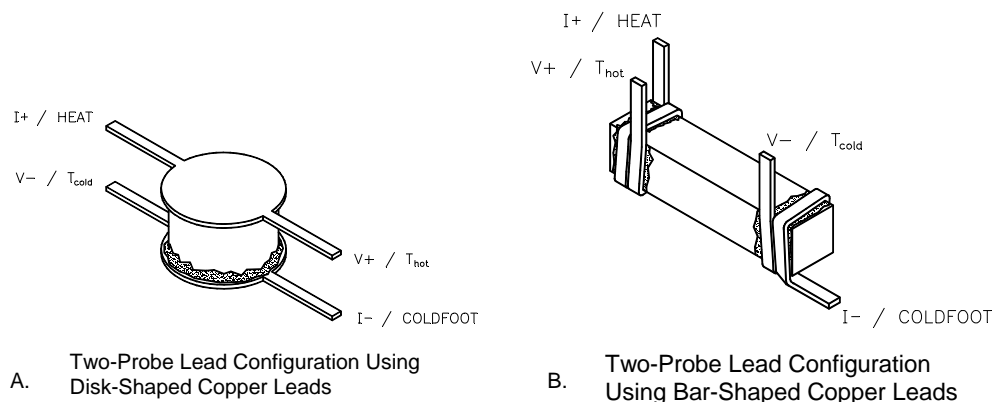


Figure 4-1. Examples of Leads Mounted in Two-Probe Configuration

Note that the thermal conductance of the epoxies decreases very rapidly below 100 K, so the thermal contact resistance may be significant at low temperature even if it is not at room temperature. If you know the cross-sectional area and the approximate thickness of the epoxy used, then you can estimate the contact resistance due to the thermal resistance of the epoxy using the data in Table 4-2 and the equation for the thermal resistance:

$$1/K = 1/\kappa \times l/A,$$

where A is the cross-sectional area of the bond and l is the thickness of the epoxy in the bond.

Table 4-2. Approximate thermal conductance of epoxies

TEMPERATURE (K)	SILVER-FILLED H20E EPOXY κ (W/m - K)	TRA-BOND 816 H01 EPOXY κ (W/m - K)
300	2	1
100	1	0.5
30	0.5	0.2
10	0.2	0.09
5	0.1	0.03
2	0.03	0.01

4.3.2 Four-Probe Lead Configuration

Use a four-probe lead configuration method when sample resistivity (thermal or electrical) is too low to allow you to neglect the contribution of lead contact resistance. Thus, the four probes are attached individually and you avoid the problem of contact resistance at the $T_{\text{hot}}/V+$ and $T_{\text{cold}}/V-$ probes. This is because very little thermal or electrical current passes into the copper shoes from the sample and hence they approximate much better the ideal of passive probes of the sample's temperature and voltage profile. Note that care must be taken to keep separate the epoxy pads on the sample, or else this advantage is compromised due to thermal/electrical currents that may shunt through the epoxy pads at the T/V probes. Figure 4-2 shows a sample mounted in the four-probe configuration.

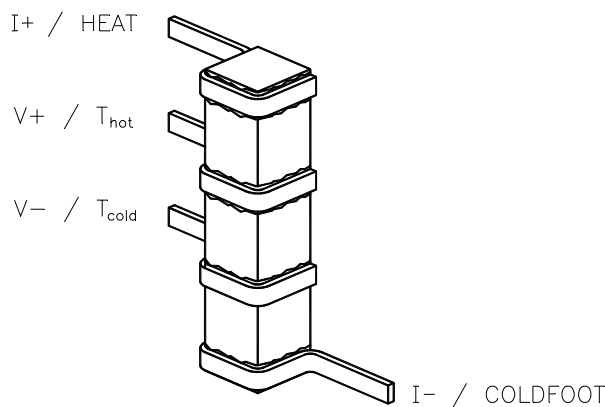


Figure 4-2. Example of Leads Mounted in Four-Probe Configuration

4.4 Checking the Sample Contact

If electrical resistivity or thermopower measurements will be performed on a sample, electrical contact to the sample must be checked after mounting it on the puck. Plug the puck into the puck-wiring test station and check contact at V+/- with an ohmmeter (use the Thermal Transport overlay on the puck box). For resistivity, the contacts at I+/- must also be checked. Finally, check that none of these leads are shorted to the puck body because this may introduce noise into the electrical measurements.

4.5 Using the Puck-Mounting Station

A puck-mounting station (Figure 2-2) is included in the user's kit to make mounting samples on the Thermal Transport puck more convenient. The heavy steel base provides stability while the puck is mounted in the plastic socket, and you can tighten the thumbscrews on the station once you have set the desired orientation of the puck. The plastic socket also acts as a gauge of the copper contact finger positioning on the puck: If the puck is not held in the socket or if it cannot fit in the socket, you must set the finger position with the puck finger adjustment tool (see Section 7.2).

Measurements

5.1 Introduction

This chapter contains the following information:

- Section 5.2 explains how to take measurements with the TTO system.
- Section 5.3 discusses the measurement mode parameters.
- Section 5.4 discusses the TTO measurement process.

5.2 Taking Thermal Transport Measurements



Thermal Transport measurements can be taken only if (a) the Thermal Transport connection cable is plugged into the gray, color-coded Lemo port on the PPMS probe head and (b) the Thermal Transport option is activated in PPMS MultiVu. Refer to Appendix A to install and activate the Thermal Transport option.



You are encouraged to use the Thermal Transport control center to perform all normal TTO system operations. The automated routines in the control center help ensure that you complete the necessary procedures when you install new samples and create data files. The examples of immediate-mode measurements in this chapter illustrate use of the control center.

5.2.1 Connect Leads to the Sample

Four leads must always be connected to any sample that will be measured with the TTO system: a source (current I+ and heat source), two probes ($T_{\text{hot}}/V+$ and $T_{\text{cold}}/V-$), and a sink (current I– and heat sink). Leads are designed to mount permanently on the sample and offer high thermal conductance and electrical conductivity from the gold-plated copper shoes to the sample.

Complete the following steps to connect leads to the sample:

1. Prepare a sample in which the estimated room-temperature thermal conductance is $K \sim 10 \text{ mW/K}$ (for best results, stay between 5 mW/K and 15 mW/K).
2. Decide whether to attach the leads in a two-probe or four-probe configuration. Refer to Section 4.3 for more information.

For leads, both bars and disks of gold-plated copper are included in the sample-mounting kit. If you use another lead material, it must have high thermal conductance yet be smaller than 0.5 mm ($.020 \text{ inch}$) in diameter in order to fit in the shoe assemblies. In addition, it must have good electrical conductivity and be free of insulating oxidized surfaces where contact is made to the shoe assemblies or to the sample.

3. Decide which epoxy material—silver-filled or insulating—is appropriate to the measurements you will take. Refer to Section 4.2.2 for more information.
4. Prepare the leads by bending them to shape so that they fit snugly around the sample to maximize thermal conductance at the contact to the sample.
5. Mount and epoxy the leads and cure them appropriately.

5.2.2 Measure the Sample Dimensions

1. Measure the length l between the hot and cold thermometer probes as well as the cross-sectional area A of the sample in the region between these probes.
2. Calculate the total surface area of the sample and leads as well as an estimate of the sample's infrared emissivity ϵ . This is necessary only for thermal conductivity measurements where some heater power is lost at high T ($> 300 \text{ K}$) to radiative thermal conduction from the hot end of the sample to the surrounding isothermal shield. If the infrared emissivity of the sample is not known, you can often employ these crude approximations:

- For nonmetallic surfaces—for example, ceramics and heavily oxidized metals— $\epsilon \sim 1$
- For unpolished metallic surfaces, $\epsilon \sim 0.3$
- For highly polished metallic surfaces, $\epsilon \sim 0.1$

If thermal radiation from the sample is a concern, you can minimize it by reducing the sample surface area or coating the sample with a thin film of known emissivity, such as varnish—assuming this does not affect other physical properties of interest to you. The infrared emissivities of several substances (for example, some metals, oxides, and paints) are tabulated in the *CRC Handbook of Chemistry and Physics*.



NOTE

An artifact from thermal radiation can be seen as T^3 “tail” in the thermal conductance that is visible at temperatures above $\sim 200 \text{ K}$. Radiation from the sample and the shoe assemblies is corrected for in the data from the Conductance (W/K) and Conductivity ($\text{W/m} \cdot \text{K}$) columns in the data file. However, no corrections are made in the Raw Conductance (W/K) data. Section 1.5.5 explains how to correct for heat losses in TTO measurements.

5.2.3 Mount the Sample

1. Place the puck in the Thermal Transport puck-mounting station and clamp the I-/coldfoot sample lead to the coldfoot on the puck by using the small Phillips screwdriver included in the user's kit.
2. Affix the shoes to the three remaining leads consistent with Figure 4-1 or 4-2 by using the small slotted screwdriver and tweezers to hold the probe shoes. Use enough force to make the M1 screws snug, but do not overtighten them because overtightening damages the soft copper of the probe shoe. Note that the white connector plug on the $T_{\text{hot}}/V+$ shoe assembly plugs into the puck at the middle socket which is painted red, while $T_{\text{cold}}/V-$ plugs into the blue socket. As a convenience in sample mounting, you may use the red and blue Sharpie permanent markers included with TTO to color the white surfaces on both ends of the hot and cold thermometer shoe assemblies.
- * Note: When attaching the heater shoe, make sure the lead does not touch the heater resistor's solder pad, potentially causing an electrical short.
3. Note the serial numbers of each of the three shoe assemblies. These numbers are handwritten on one side of the connector plug at the puck.



While attaching the shoes, use care not to pull on the thin wires running to the probe shoes, and avoid scratching the areas near solder pads, especially where the wire is soldered to the back wall of a shoe.

4. Check carefully that neither the shoes nor their wires are touching each other or any part of the puck, and that the sample is contacting the puck only at the clamp of the coldfoot.
5. Place the radiation shield on the puck, remove the shield cap, and inspect to verify that no wires, shoes, or that the sample touches the shield.
6. Replace the shield cap before you insert the sample into the PPMS probe.

5.2.4 Install the Sample

1. Activate the Thermal Transport option if the option is not currently active. Do the following: (a) select **Utilities** > **Activate Option** in the PPMS MultiVu interface, (b) click on **Thermal Transport** under the **Available Options** heading, and then (c) select **Activate**.
2. Select the **Install** tab in the Thermal Transport control center (Figure 3-2).
3. Select **Install Wizard** and follow the software prompts to install the sample in the PPMS sample chamber.

5.2.5 Start the High-Vacuum System

If the install wizard did not start the high-vacuum system, you start the high-vacuum system by doing one of the following: either (a) select the **HiVac** button in the **Install** tab in the Thermal Transport control center or (b) select **Instrument** > **Chamber** > **HiVac** in the main PPMS MultiVu interface. Wait for the HiVac state to be reached before starting thermal transport measurements, because a significant heat leak can result from gas thermal conduction away from the TTO heater.

5.2.6 Open the Data File

1. Select the **Data File** tab in the Thermal Transport control center (Figure 3-3). The tab indicates which data files are selected to save the measurement data. If you run the measurement when no data file is selected, the data is discarded.
2. Click **Browse** to select a different file or create a new file. **Browse** opens the **Thermal Transport Select Data File** dialog box, which lists all existing files.
3. Select a data file or enter the name of a new file. When you create a file, the software prompts you to define the sample properties for the sample whose measurement data will be saved to the file, and the data entry fields in the control center's **Sample** tab are enabled. Define the sample properties, and then select **OK**.

As soon as you select or create a file, the **Data File** tab appears again, and the **File Name** field identifies the data file you have selected. Notice that by default the **Capture Raw Data** check box is *not* selected. If this box is enabled, raw measurement data is saved to a raw data file that has the same name as the measurement data file but a **.raw** file extension instead of a **.dat** file extension.

5.2.7 Define the Measurement

1. Decide which mode—continuous or single—to use for the measurement. Continuous mode is the software default and generally offers the most rapid data acquisition. Single mode offers the most direct control of the measurement. To specify single mode measurements, use the **Mode** tab of the **Thermal Transport Measurement** dialog (Figure 3-10). Single mode measurements may be steady state or timed. Refer to Section 5.4 for more details about each measurement.
2. Decide whether to take the measurements directly by accessing the **Thermal Transport Measurement** dialog (select the **Measure...** button in the control center) or to run the measurements in a PPMS MultiVu sequence.
3. Select the **Measure...** button in the control center so that you see the **Settings** tab in the **Thermal Transport Measurement** dialog (Figure 3-7). You use the **Settings** tab to decide which physical quantities you want to measure: thermal conductivity, Seebeck coefficient, electrical resistivity, and (if all the above are selected) whether you would like the program to compute the figure of merit ZT from these measurements and put this quantity in the data file.
4. Define appropriate parameters for each measurement. The default values for these parameters are appropriate for most cases, so it is unlikely you will need to change them.
 - In continuous mode, which uses an adaptive algorithm, you must set the allowable ranges for thermal measurement parameters. These parameters, which include measurement period, heater power, and Seebeck voltage, are set in the **Thermal** tab in the **Thermal Transport Measurement** dialog (Figure 5-1). Similarly, for resistivity measurements you must define the range of excitation and frequency parameters. You set these parameters in the **Resistivity** tab in the **Thermal Transport Measurement** dialog (Figure 5-2).
 - In single mode, you state the style of single mode measurement along with the fixed heater power, measurement period, and maximum expected Seebeck voltage.

5.2.8 Run the Measurement

5.2.8.1 RUNNING THE MEASUREMENT INTERACTIVELY

To run a thermal transport measurement interactively, you select the **Measure...** button that is at the bottom of the Thermal Transport control center. Selecting the **Measure...** button opens the **Thermal Transport Measurement** dialog box, which you use to select the thermal properties you want to measure as well as the parameters and limits for the measurements. By default, dynamic (continuous) measurements are made using an AC technique unless you use the **Mode** tab (Figure 3-10) to switch to steady-state (single) measurements. Refer to Section 1.3 for more information on the various modes of operation. The measurement does not run until you select the **Start** button in the **Thermal Transport Measurement** dialog.

Data from any measurement is automatically saved if a data file is open.

5.2.8.2 RUNNING THE MEASUREMENT IN A SEQUENCE

To run a measurement in sequence mode, you run a sequence that contains **TTO Measure** commands (under **Measurement Commands**). The measurement is taken automatically when PPMS MultiVu reads the measurement command in the running sequence.

Thermal measurements in sequence mode can generally be taken in two ways:

- Slowly ramp the temperature (rate ~ 0.5 K/min.) and measure continuously. This method uses the **Set Temperature** sequence command and is usually the more expedient technique.
- Scan in temperature (or magnetic field) and take single measurements after the system has stabilized at the new temperature or field. This method uses the **Scan Temperature/Scan Field** commands.

Data from any measurement is automatically saved if a data file is open.

The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses sequence files and all standard system sequence commands in detail.

5.2.9 Scanning or Ramping the Temperature While Measuring

In either interactive or sequence mode, the most common measurement technique is to start continuous measuring and then ramp the temperature slowly by using the **Set Temperature** command with a slow rate (slew rates are typically ~ 0.1 – 1 K/min.). For example, if the system is at room temperature, you can set a target temperature of 1.8 K and a slew rate of 0.5 K/min.

If you are taking single steady-state measurements, these are obviously taken at a fixed temperature and field. This method is more amenable to the **Scan Temperature** command, which allows you to wait for system stability at each target before taking a measurement.

5.3 Measurement Mode Parameters

5.3.1 Continuous Measurement Mode

Parameters for continuous mode measurements are defined in the **Thermal** tab in the **Thermal Transport Measurement** dialog box. You open this dialog box by selecting the **Measure...** button in the Thermal Transport control center.

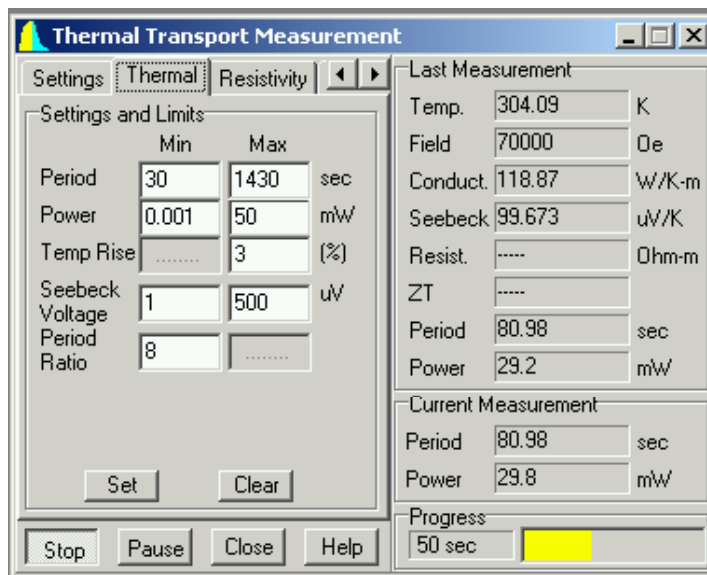


Figure 5-1. Thermal Tab in Thermal Transport Measurement Dialog Box

The **Clear** button in the **Thermal** tab restores either the values that were in memory after the last time the **Set** button in the tab was selected, or the default values if **Set** has not yet been selected.

Table 5-1. Minimum and Maximum Parameter Limits for Continuous Mode Measurements

PARAMETER	FUNCTION	DESCRIPTION
Period	Length, in seconds, of heater on/off cycle.	Minimum is 30 seconds due to limited data acquisition rate of hot/cold Cernox temperatures from user bridge. Maximum period is 1430 seconds. Only discrete values of the period are allowed: period = $4292/n$, where n is an integer ($n > 2$). This implies that jumps between available periods become larger as period grows.
Power	Limits power that heater resistor can output. Expressed in mW.	Default limits are 1 μ W and 50 mW. Minimum value is limited by hardware limitations of the DAC of the current source to 10 μ A, which translates to 0.2 μ W for 2-k Ω heater. Maximum value is set by voltage compliance of 10 V of the current source, which translates to 50 mW for 2-k Ω heater.

(continues)

Table 5-1. Minimum and Maximum Parameter Limits for Continuous Mode Measurements (Continued)

PARAMETER	FUNCTION	DESCRIPTION
Temp. Rise	Target value of temperature rise during heating, expressed in percent of absolute temperature. Heater power is adaptively adjusted to achieve the desired Temp. Rise.	Defined as rise in T_{hot} due to the heater, divided by average temperature during this time. This parameter can be decreased in order to minimize uncertainty in temperature during a pulse, such as in cases where physical properties are changing very rapidly with temperature. Errors in data increase sharply if Temp. Rise < 1% is chosen, due to small magnitude of thermal signal.
Seebeck Voltage	Determines initial gain settings, in μV , for DSP voltage readback preamp.	Entering an expected maximum value determines initial gain settings for DSP voltage readback preamp. Software uses “sticky autorange” algorithm to rescale preamp if initial guess was far off.
Period Ratio	Provides feedback for heater period.	Defined as ratio of the period to long time-constant τ_1 of sample. Default period ratio value of 8 has been found empirically to be near minimum needed by curve-fitting algorithm in order to converge on correct result.

Table 5-2. General Settings for Continuous Mode Measurements

PARAMETER	FUNCTION
Save Marginal Results	Prompts software to write to data file results for which fitting algorithm was able to converge on a value but encountered significant errors from one or more sources. Results are deemed marginal if the error on the curve fit is between 50% and 200%.
Discard First N Results	Prompts software to discard user-specified number of first results. This setting is useful because first several (~ 3) data points are usually of poor quality because parameters were still in the process of being optimized.

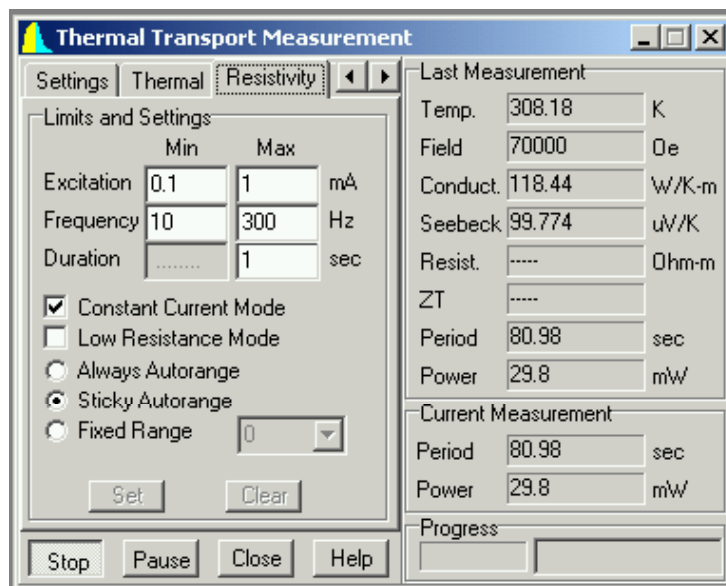


Figure 5-2. Resistivity Tab in Thermal Transport Measurement Dialog Box

Table 5-3. Resistivity Excitation Parameters for Continuous Mode Measurements

PARAMETER	FUNCTION
Excitation	Defines minimum and maximum excitation amplitude in milliamps.
Frequency	Defines minimum and maximum measurement frequency in Hz.
Duration	Defines measurement duration in seconds.



NOTE

The parameters for resistivity measurements are adjusted adaptively in the continuous measurement mode, in the same way as the heater power and period are allowed to vary. Refer to the *Physical Property Measurement System: AC Transport Option User's Manual* for resistivity settings.

5.3.2 Single Measurement Mode

The parameters for single mode are generally simpler than the parameters for continuous mode. Basically, you fix the heater power and set a heater on/off criterion that depends on the style of measurement. In **stability** mode, the criterion is the temperature stability on both the hot and cold thermometer probes. In **timed** mode, you set a period length for the heater pulse that can be as long as desired. Finally, for **Seebeck voltage** you enter an estimate of the change in thermal voltage (in μV) that you expect from the sample during the measurement. This determines the gain settings on the DSP circuitry in the AC Transport controller.

5.4 Description of Measurement Process

With the TTO system you can measure thermal properties in either continuous mode or single mode. The conceptual distinction between the two is that in continuous mode, the system is constantly measuring the thermal properties and any acceptable data is saved to the data file, while in single mode the system takes a measurement only when you request that it do so. An advantage of the continuous technique is that a higher density of data is obtained, and you take advantage of the adaptive algorithms built into the software which are constantly optimizing the system parameters for each measurement. However, it may be preferable in some cases to have more direct control of the measurement—for example when one measurement takes several minutes or when the adaptive software routines may not adequately adjust to rapidly changing thermal transport properties of the sample—and in these cases you want to use single mode.

In continuous measurement mode, the parameters of heater power and period as well as the resistivity excitation and frequency are continually being updated after each heater pulse to keep the Temp. Rise and Period Ratio parameters near the user-set values. The raw data $\Delta T(\text{time})$ is sent to the fitting algorithm, which performs a three-parameter, nonlinear, least-squares fit in ΔT_∞ , the asymptotic temperature drop across the sample, as well as τ_1 and τ_2 , which are a long and short time constant, respectively, that characterize the sample-lead-shoe thermal circuit. Of these, ΔT_∞ is used to calculate the thermal conductance of the sample $K = (\text{heater power}) / \Delta T_\infty$, while the period for the next measurement is computed from τ_1 by $\text{Period} = \text{PeriodRatio} \times \tau_1$. The asymptotic Seebeck voltage ΔV_∞ is computed from the raw data $\Delta V(\text{time})$ similarly, except that a computationally simpler linear regression is used because τ_1 and τ_2 are based on the conductivity fitter routine. The Seebeck coefficient is then simply $\alpha = \Delta V_\infty / \Delta T_\infty$. Resistivity measurements are made before and after the heater pulse, and the average of the two is taken as the sample resistivity. If the thermoelectric figure of merit is sought, then $ZT = \alpha^2 \times T / (\kappa \times \rho)$ is computed and written to the data file.

Single measurements, which are generally most useful to advanced users with specialized requirements, are subdivided into stability and timed measurement methods. In the stability technique, you specify requirements for thermal stability at the hot and cold thermometer probes on the sample and a user-defined heater power is applied only after the stability criterion is met. Temperature and/or voltage data points are taken in the quiescent state before the heater is turned on, and heater power is applied until thermal stability is once again achieved at both thermometer probes, at which point the second series of data points is taken. In a timed measurement, you set a fixed period and power for one heater pulse. Note that in all these single-mode thermal measurements, data points are taken only at the end of the settling time and at the end of the heater “on” segment, and it is assumed that $\Delta T = \Delta T_{\text{on}} - \Delta T_{\text{off}}$ and $\Delta V = \Delta V_{\text{on}} - \Delta V_{\text{off}}$ represent asymptotic steady-state values; that is, no curve fitting of the data is performed.

See Section 1.5 for more information on the theory of operation for the Thermal Transport option.

Troubleshooting

6.1 Introduction

This chapter contains the following information:

- Section 6.2 offers suggestions to help troubleshoot jumps or noise in TTO data.
- Section 6.3 discusses ways to minimize errors due to radiation effects.
- Section 6.4 refers to high-vacuum problems.

6.2 Jumps or Noise in the Data

The following are some general guidelines for ensuring good data quality:

1. Make sure that the condition Period Ratio ($= \text{Period}/\tau_1$) > 8 is met at all temperatures. Considering that the maximum period is 1430 seconds, your sample must be designed so that the thermal diffusion time τ_1 is not too long. If Period Ratio is too small, the curve-fitting software is not able to adequately fit the data.
2. Make sure that an adequate heat pulse can be applied (default is Temp. Rise = 3%) across the sample; that is, the thermal conductance of the sample is not above about 20 mW/K. If Temp. Rise falls below the 1% level, data can become noisy.
3. Verify that the leads are attached to the sample with a generous amount of epoxy that is well cured and that the lengths of the leads are kept to a minimum.
4. If temperature or field is being slewed while measurements are made, verify that the slew rate is slow (typically less than 1 K/min.) and uniform over the course of a measurement.

6.2.1 Gaps in the Data Versus Temperature

- Check that the background temperature slewing of the system as reflected in the hot and cold sample thermometers and the system thermometer (see the `.raw` data file) was linear and not irregular.
- Check the value for the period of thermal measurements in the vicinity of the gap, because a very long period results in lower density of data.
- Look in the **TTO Error Count** and **TTO Status Log** dialogs for evidence of errors. Also check the codes for both the PPMS (**Status**) and TTO (**Error**) status in the data file and consult Appendix B to interpret the codes.

6.2.2 Steps in the Data

If the data jumps in a step-wise fashion as a function of temperature or field, do the following:

- Check to see if the jump exists only in the magnitudes of thermal conductance and electrical resistance, but is absent in Seebeck coefficient data. This is evidence that the sample geometry (A/l) has changed due to internal cracking of the sample (this can often occur under temperature cycling) or breaking of the epoxy bonds to the leads. Weak epoxy bonds are evidence of poor epoxy strength or poor thermal matching of the epoxy and the sample.
- Look at the standard deviation in the measured quantities and also at the `.raw` data to see if the software is adequately modeling the data. A step in the data can occur when the data fitting is poor enough that several distinct solutions have comparable curve fit errors. See the **Error** column in the `.dat` file and consult Appendix B to interpret the TTO status code.

6.3 Thermal Radiation “Tail” in the Thermal Conductivity Data

Thermal radiation between the sample (and shoe assemblies) and the surrounding environment introduces errors in the measurement of thermal conductivity at high temperatures. Some of the heat produced by the heater resistor radiates instead of traveling through the sample, in accordance with the radiation law described in Section 1.5.5. The resulting “tail” has a T^3 temperature dependence that is generally observable only above about 200 K. Described here are some ways of minimizing errors due to radiation effects. You may try one or more of these techniques in order to manage thermal radiation in your measurements:

1. Increase the geometrical factor of the sample A/l so as to make the thermal conductance of the sample much higher than the errors associated with subtracting the radiation thermal conductance. These errors are about ± 1 mW/K at the highest temperatures.
2. Minimize errors in estimating sample radiation by coating the surface of the sample with a material of known emissivity, or by choosing a sample geometry minimizing the radiation surface area.
3. Modify the mounting of leads on the sample by thermally sinking the heater and hot thermometer shoes together, and connect only the heater to a lead on the sample. The shoes can be affixed to each other by using H Grease to stick them together back-to-back, and by making sure that they do not tend to separate. When the shoes are placed in this special configuration, it is recommended that you take measurements in single mode because the fitter algorithm used in continuous mode often does not adequately model this thermal circuit. The reason this technique works is that the stray conductance due to the shoe assemblies, K_{shoes} , was estimated in a configuration in which the heater and hot thermometer were isothermal. However, in real samples the heater will always be hotter than the hot thermometer so that some thermal radiation from the heater is not entirely accounted for.

6.4 High-Vacuum Problems

For help troubleshooting the PPMS High-Vacuum option, please refer to the appropriate manual, either the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* or the *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*, depending on the type of high-vacuum system that you have.

Maintenance

7.1 Introduction

This chapter contains the following information:

- Section 7.2 explains how to use the puck adjustment tool.
- Section 7.3 explains how to grease the puck fingers and the coldfoot clamp.

7.2 Using the Puck Adjustment Tool

The puck adjustment tool (Figure 7-1) adjusts the tension in the chuck fingers so that the fingers maintain solid thermal contact with the heater block located at the bottom of the sample chamber. Solid thermal contact between the chuck fingers and the heater block is especially important for high-vacuum applications, such as heat capacity and thermal transport measurements.

The puck adjustment tool consists of two metal cylinders. In Figure 7-1, cylinder 1 is the finger spreader, and cylinder 2 is the finger contractor and the test cutout. The finger spreader and the finger contractor adjust the tension of the chuck fingers. The test cutout, which has the same dimensions as the cutout in the heater block, tests how well the chuck fingers will contact the heater block.

You use the puck adjustment tool on the puck after you have inserted the puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the sample chamber.

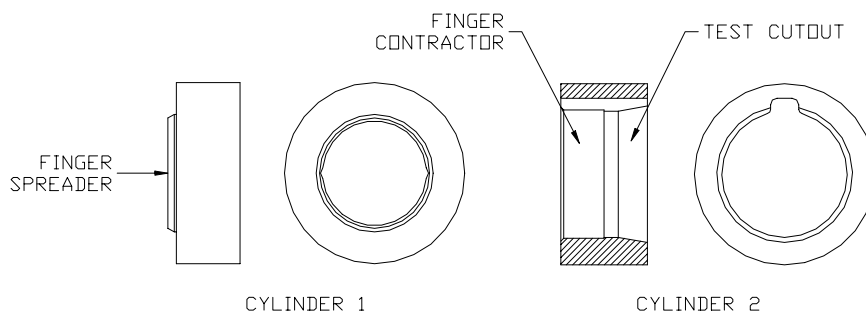


Figure 7-1. Puck Adjustment Tool

Complete the following steps to use the puck adjustment tool:

1. Screw the thermal radiation shield onto the TTO puck.
2. Place the puck on the finger spreader. Refer to Figure 7-1. Turn the puck until the screw heads on the bottom of the puck line up with the grooves inside the finger spreader. Press the puck downward and continue pressing until all chuck fingers touch the base of the finger spreader. When all fingers touch the base of the spreader, the spreader evenly applies radial force to the fingers, pushing them outward and slightly beyond their optimal location.
3. Remove the puck from the finger spreader.
4. Place the puck inside the finger contractor. Refer to Figure 7-1. Press straight down on the puck and continue pressing until you press the puck completely into the finger contractor. When the entire chuck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that the fingers obtain their optimal location.
5. Remove the puck from the finger contractor.
6. Place the puck inside the test cutout. Refer to Figure 7-1. Verify that the puck fits easily but snugly in the test cutout.

7.3 Greasing the Puck Fingers and the Coldfoot Clamp

The thermal contact between the sample puck and the heater block can be further improved by applying a small amount of H Grease (which is included in the TTO user's kit) to the puck fingers. You may also wish to use a small amount of H Grease on the sample's cold lead when clamping it in the coldfoot, because this contact can be a significant thermal resistance. Note that it is always important to apply enough tension to the coldfoot screw in order to have good thermal and electrical contact between the sample lead and the coldfoot.

Installation

A.1 Introduction

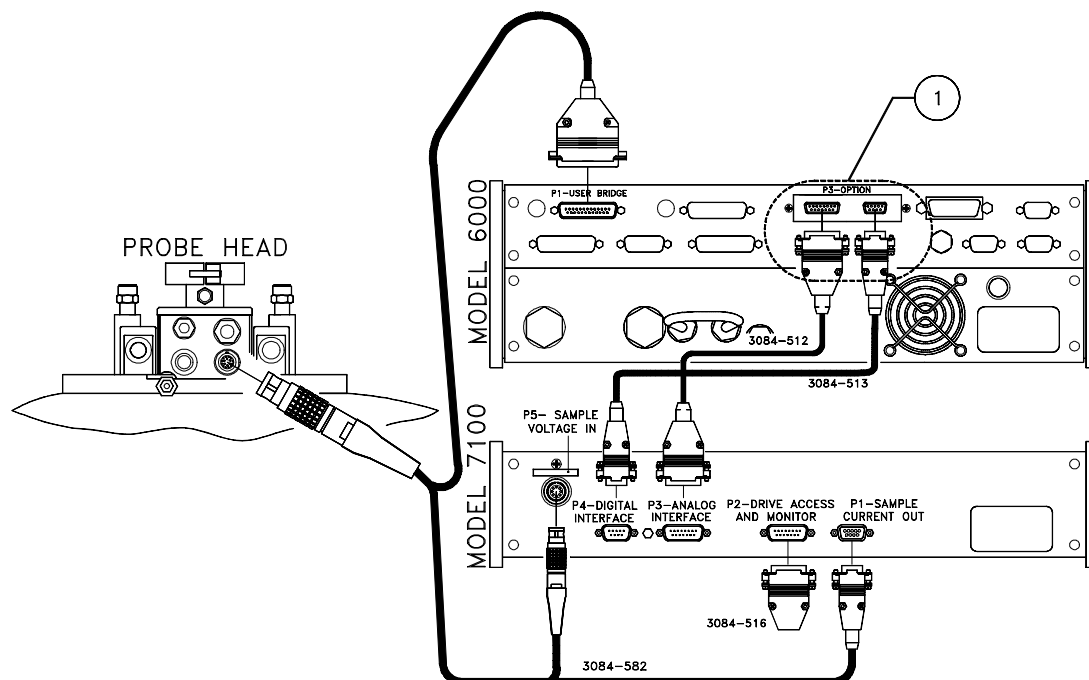
This appendix contains the following information:

- Section A.2 explains how to install the TTO hardware.
- Section A.3 explains how to install the TTO software.

A.2 Installing Thermal Transport Hardware

To install the TTO hardware, you must install all components that are necessary for the TTO system. These components may include the following:

- **Model 7100 AC Transport Controller**
For more information about the Model 7100, refer to section 2.3.1 in this manual and to the *Physical Property Measurement System: AC Transport Option User's Manual*.
- **High-Vacuum option**
The TTO system works with either the Turbo Pump High-Vacuum option or the Cryopump High-Vacuum option. For more information about these options, refer to the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* or the *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*.
- **User bridge board**
For more information about the user bridge board, refer to section 2.2.6 in this manual and to the *Physical Property Measurement System: Resistivity Option User's Manual*.
- **Thermal Transport connection cable**
This cable must be connected as described in section 2.2.5 and as illustrated in Figure A-1 on the following page.
- **WaveROM EPROM (certain systems only)**
If the ACMS waveROM upgrade kit was included with your TTO system, you must install the new ROM. Refer to sections 2.2.4 and 2.2.4.1.



① THESE CABLES MAY GO TO THE OPTION CONTROLLER
DEPENDING ON SYSTEM CONFIGURATION.

Figure A-1. Thermal Transport Option Connection Diagram

A.3 Installing Thermal Transport Software

1. Install the PPMS MultiVu software (version 1.1.6 or later) if it is not already installed. Do the following: (a) insert PPMS MultiVu Disk 1 into the PC, (b) select the A: drive, (c) select `setup.exe`, and then (d) complete all operations the InstallShield wizard prompts you to perform.

The TTO software runs in conjunction with the PPMS MultiVu software. PPMS MultiVu must be installed on the host computer in order for the TTO software to work. If you try to install the TTO software before you install PPMS MultiVu, the InstallShield wizard for the TTO software fails and generates a warning message, which tells you to install PPMS MultiVu.

2. Install the TTO software. Do the following: (a) insert Disk 1 for the TTO software into the PC, (b) select the A: drive, (c) select `setup.exe`, and then (d) complete all operations the InstallShield wizard prompts you to perform.
3. Activate the Thermal Transport option in PPMS MultiVu. Do the following: (a) start up PPMS MultiVu, (b) select **Utilities**►**Activate Option**, (c) click on **Thermal Transport** under the **Available Options** heading, and then (d) select the **Activate** button.

As soon as you activate the Thermal Transport option, the Thermal Transport control center opens and the **Measure** menu items and measurement sequence commands that are specific to TTO appear in the PPMS MultiVu interface.



Note that in order to run, TTO requires that the AC Transport (ACT) option be installed because TTO and ACT share the hardware configuration file

C:\QdPpms\ACTrans\Calibration\Actcal.cfg. For information about installing the ACT option, refer to the *Physical Property Measurement System: AC Transport Option User's Manual*.

Status Codes and Error Messages

B.1 Introduction

This appendix contains the following information:

- Section B.2 lists the system status codes for the PPMS and for TTO.

B.2 System Status Codes

B.2.1 General PPMS System Status Codes

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes

BITS	VALUE	TEMPERATURE STATUS
0–3	0	Status unknown.
	1	Normal stability at target temperature.
	2	Stable.
	5	Within tolerance; waiting for equilibrium.
	6	Temperature not in tolerance, not valid.
	7	Filling/emptying reservoir.
	10	Standby mode invoked.
	13	Temperature control disabled.
	14	Request cannot complete, impedance not functioning.
	15	General failure in temperature system; contact Quantum Design.

(continues)

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes
(Continued)

BITS	VALUE	MAGNET STATUS
4–7	0	Status unknown.
	1	Persistent mode, stable.
	2	Persist switch warming.
	3	Persist switch cooling.
	4	Driven mode, stable at final field.
	5	Driven mode, final approach.
	6	Charging magnet at specified voltage.
	7	Discharging magnet.
	8	Magnet reset.
	9	Current error, incorrect current in magnet.
	10	Persistent switch heater error.
	11	Magnet quench.
	12	Magnet charging error.
	14	Power supply error.
	15	General failure in magnet control system.
BITS	VALUE	CHAMBER STATUS
8–11	0	Status unknown.
	1	Purged and sealed.
	2	Vented and sealed.
	3	Sealed, condition unknown.
	4	Performing purge/seal routine.
	5	Performing vent/seal sequence.
	6	Pre-pump (turbo pump) / High vacuum evacuate (cryopump).
	7	High vacuum.
	8	Pumping continuously.
	9	Pre-vent / Flooding continuously.
	14	High vacuum error.
	15	General failure in gas control system.

(continues)

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes
(Continued)

BITS	VALUE	SAMPLE POSITION STATUS
12–15	0	Status unknown.
	1	Sample stopped at target value.
	5	Sample moving toward set point.
	8	Sample hit limit switch.
	9	Sample hit index switch.
	15	General failure.

B.2.2 Thermal Transport System Status Codes

Table B-2. TTO Status Codes

ERROR	BIT FIELDS
Computation : poor curve fit	1–5
Computation : no curve fit	6–10
Reserved	11–16
Fatal Error : software	17–21
Fatal Error : hardware	22–26
Reserved	27–32

In Table B-2, “poor curve fit” means that the residual of the fit was greater than 50% or that the Period Ratio was less than 4, and “no curve fit” means that the software was unable to fit the data to the model.

The five bits for each error category represent, respectively, errors in thermal conductivity, Seebeck coefficient, first resistivity, second resistivity, or figure of merit. At a glance, you can interpret the TTO status code as the following:

code = 0	No errors in measurements or computations
1 < code < 32	One or more poor computed curve fits
33 < code < 1024	One or more failed curve fits
code > 1024	Fatal error

Pinout Tables

C.1 Introduction

This appendix contains the following information:

- Section C.2 discusses and illustrates the pinouts for the TTO system.

C.2 Thermal Transport Pinouts

The following table and diagram detail the pinouts for each connector in the TTO system. The diagram illustrates hardware ports, not connectors at the end of the cables.

C.2.1 Sample Connections

The Thermal Transport connection cable (Figure 2-5) has connections to both the Model 7100 AC Transport controller and the Model 6000 user bridge board. As in the case for the AC Transport (ACT) option, connections from the Model 7100 to the sample are configured to minimize “cross-talk” between the excitation signal (the P1 port on the Model 7100) and the detected signal (the P5 port on the Model 7100). In addition, each “+/-” pair is individually twisted in the gray Lemo cable and also in the wiring between the puck base and the sample. The exceptions to this are the I+/- leads and the V+/- leads between the five-pin connector plugs and the sample (recall that V+ and V- run to separate shoe assemblies, so they are physically separated at this point).

Table C-1 on the following page lists all TTO sample connections. Figure C-1 illustrates the sample connections, and Figure C-2 illustrates the pinout of the connector sockets on the Thermal Transport sample puck.

Table C-1. TTO Sample Connections

FUNCTION	SYMBOL	SAMPLE PUCK / GREY LEMO	P1 PORT ON MODEL 7100	P5 PORT ON MODEL 7100	P1 USER PORT ON MODEL 6000
Current + (thermometers)	I_{Th+}	3			5 + 7
Current – (thermometers)	I_{Th-}	4			18 + 20
Current + (heater)	$Q+$	5	1		
Current – (heater)	$Q-$	6	6		
Voltage + (cold thermometer)	V_{Th}^{C+}	7			8
Voltage – (cold thermometer)	V_{Th}^{C-}	8			21
Voltage + (warm thermometer)	V_{Th}^{W+}	9			6
Voltage – (warm thermometer)	V_{Th}^{W-}	10			19
Current + (sample)	$I+$	11	2		
Current – (sample)	$I-$	12	7		
Voltage + (sample)	$V+$	13		1 + 3 + 5 + 7	
Voltage – (sample)	$V-$	14		2 + 4 + 6 + 8	
Ground			5	shell	13

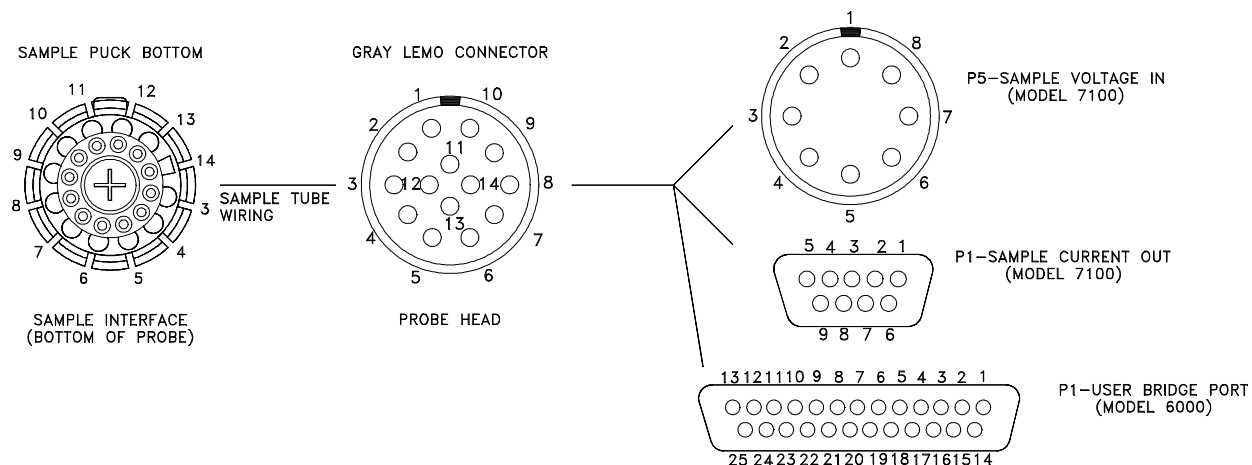


Figure C-1. Illustration of TTO Sample Connections, Showing Hardware Ports

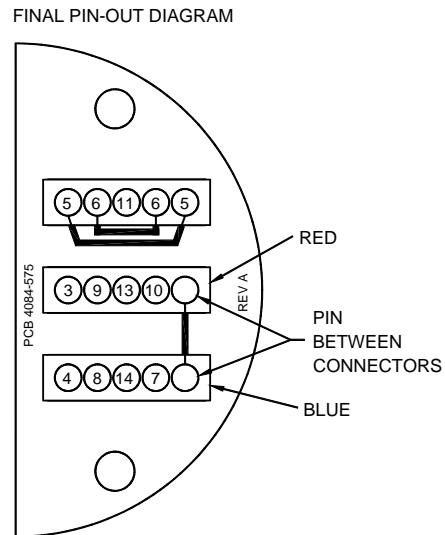


Figure C-2. Top View of Pinout of Connector Sockets on Thermal Transport Sample Puck

References

- Maldonado, O. 1992. Pulse method for simultaneous measurement of electric thermopower and heat conductivity at low temperatures. *Cryogenics* 32:10, 908–12.
- Quantum Design. 2001. *Physical Property Measurement System: AC Transport Option User's Manual*.
- . 2000. *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*.
- . 2000. *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.
- . 1999. *Physical Property Measurement System: Resistivity Option User's Manual*.
- . 2000. *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual*.
- Weast, R. C. 1988. *CRC Handbook of Chemistry and Physics*. Boca Raton, FL: CRC Press.

Index

- AC board
 - function, 2-7
 - housing WaveROM, 2-5, 2-6
- ACT option
 - hardware, 2-7
 - required for Thermal Transport option, 1-2, A-3
- AC Transport Measurement System. *See* ACT option
- Advanced tab
 - in control center, 3-5
 - in Measurement dialog box, 3-8
- Baffle assembly, 2-8
- Calibration files, 3-2
- Calibration fixture
 - plugging into puck, 2-9
 - in user's kit, 2-3
- Charcoal holder, 2-8, 2-9
- Connection diagram, A-2
- Contact baffle, 2-8
- Continuous Low-Temperature Control option, 1-2
- Continuous measurement mode
 - parameters, 5-6–5-8
 - ramping temperature, 5-5
 - system operation, 1-3, 5-9. *See also* Thermal Transport software: Models
 - using, 5-4
- Control center. *See* Thermal Transport control center
- Cryopump. *See* High-Vacuum option
- Data files. *See* Measurement data files; Raw data files
- Data File tab, 3-3, 3-4
- Data logging dialog, accessing, 3-8
- Data, plotting in Waveform tab, 3-4
- Data, troubleshooting
 - jumps, 6-1–6-2
 - thermal radiation tail, 6-3
- Electrical resistivity
 - estimating errors, 1-9–1-10
 - how measured, 1-6, 5-9
 - measuring, 5-1–5-5. *See also* Measurements
 - parameters, 3-7, 5-8
 - raw data from measurement, 3-13
 - significance, 1-3
 - units expressing, 3-2
- Epoxy, for sample leads, 4-3
- Error Count dialog box, 3-14
- ExportData.exe program, 3-14
- Figure of merit
 - estimating errors, 1-9–1-10
 - how measured, 1-6, 5-9
 - measuring, 5-1–5-5. *See also* Measurements
 - significance, 1-3
- Four-probe lead configuration, 4-4–4-5
- Hardware
 - ACT option, 2-7
 - High-Vacuum option, 2-8
 - installing, A-1–A-2
 - Thermal Transport option, 2-1. *See also* Thermal Transport connection cable; Thermal Transport sample puck; User bridge board; User's kit; WaveROM EPROM
- Header, data file, 3-10–3-11
- Heat loss correction, 1-11
- High-Vacuum option
 - hardware, 2-8
 - required for Thermal Transport option, 1-2
 - starting, 5-3
 - troubleshooting, 6-3
- Immediate-mode measurements. *See* Interactive measurements
- Infrared emissivity, estimating, 5-2
- Installation
 - hardware, A-1–A-2
 - secondary, 1-11
 - software, A-3
- Install tab, 3-3
- Interactive measurements, 3-1, 3-5, 5-5. *See also* Measurements
- Isothermal radiation shield
 - function, 2-2
 - part number, 1-2
- Lead configurations, 4-4–4-5
- Log file. *See* Thermal Transport log
- Magnetic field range, 1-2
- Maintenance. *See* Puck adjustment tool; Puck fingers
- Measurement data files
 - active, identified in control center, 3-10
 - format, 3-11–3-12
 - header, 3-10–3-11
 - saving marginal measurement results, 3-5
 - saving PPMS system items, 3-8, 3-9
 - storing sample measurement data, 3-2, 3-10, 5-5
 - viewing, 3-14

Measurements

- defining, 5-4. *See also* Mode tab; Resistivity tab; Settings tab; Thermal tab
- modes. *See* Continuous measurement mode; Single measurement mode
- plotting data in Waveform tab, 3-4
- process, description of, 5-9
- properties measured, 1-5–1-6. *See also* Electrical resistivity; Figure of merit; Seebeck coefficient; Thermal conductivity
- standard for. *See* Nickel calibration samples
- status, shown in control center, 3-9
- storing data from. *See* Measurement data files; Raw data files
- taking, 5-1–5-5
- units expressing, 3-2

Model 7100 AC Transport Controller

- function, 2-7
- in Thermal Transport connection diagram, A-2
- sample connections, C-2

Mode, measurement. *See* Continuous measurement mode; Single measurement modeMode tab, 3-8. *See also* Continuous measurement mode; Single measurement mode

Nickel calibration samples

- parameters, 2-5
- part number, 1-2
- in user's kit, 2-3

Parameters

- continuous measurement mode, 5-6–5-8
- curve fitting results, 3-4
- nickel calibration samples, 2-5
- single measurement mode, 5-8
- system, 1-2

Part numbers, 1-2

Period ratio, recommended value for, 3-6

Pinouts, C-1–C-3

PLCC chip extraction tool, 2-5, 2-6

PPMS MultiVu

- installing, A-3
- required for Thermal Transport option, 1-2, 3-1
- viewing data files in, 3-14

Puck. *See* Thermal Transport sample puck

Puck adjustment tool

- function, 7-1
- illustration, 7-1
- using, 7-2

Puck fingers, greasing, 7-2

Puck-mounting station

- illustration, 2-3
- using, 4-5

Puck-wiring test station, 4-5

Radiation shield. *See* Isothermal radiation shield

Raw data files

- format, 3-13
- saving data to, 3-10, 5-4
- viewing, 3-14

Resistivity measurement. *See* Electrical resistivity

Resistivity option, 1-2

Resistivity tab

- continuous measurement mode parameters set in, 5-8
- defining resistivity excitation parameters, 3-7
- illustration, 3-7, 5-8

ROM. *See* WaveROM EPROM

Sample

- checking electrical contact, 4-5
- connecting leads to, 5-2
- connections, C-1–C-3
- dimensions, measuring, 5-2. *See also* Measurement data files: Header
- four-lead requirement, 4-1, 5-2
- geometry, 4-2
- heat loss correction, 1-11
- idealized, connections for, 1-7
- installing, 5-3
- lead length, 4-1
- measuring. *See* Measurements: Taking
- minimizing resistance, 4-1
- mounting, 5-3. *See also* Lead configurations; Puck-mounting station
- puck. *See* Thermal Transport sample puck
- response, diagram of, 1-8

Sample tab, 3-4

Seebeck coefficient

- estimating errors, 1-9–1-10
- how measured, 1-5, 5-9. *See also* Thermal Transport software: Models
- of manganin leads, 1-11
- measuring, 5-1–5-5. *See also* Measurements
- parameters, 3-6, 5-6–5-7
- raw data from measurement, 3-13
- significance, 1-3
- units expressing, 3-2

Sequence measurements. *See also* Measurements

- ramping temperature, 5-5
- taking, 5-5
- versus usage in other PPMS options, 3-1

Settings tab

- continuous measurement mode parameters set in, 5-7
- defining basic measurement settings, 3-5

Shoe assemblies

- calibrating new, 2-9–2-10
- description, 2-2

Single measurement mode

- parameters, 5-8
- ramping temperature, 5-5
- styles, 1-4
- system operation, 1-3–1-4, 5-9
- using, 5-4

Software. *See* PPMS MultiVu; Thermal Transport software

Status codes

- general PPMS, B-1–B-3
- Thermal Transport system, B-3

Status, system. *See* Thermal Transport control center:

- Showing system status

- Temperature
 - calibration, 2-9
 - ramping during measurements, 5-5
 - system range, 1-2
- Thermal conductivity
 - estimating errors, 1-9–1-10
 - how measured, 1-5, 5-9. *See also* Thermal Transport software: Models
 - measuring, 5-1–5-5. *See also* Measurements
 - parameters, 3-6, 5-6–5-7
 - raw data from measurement, 3-13
 - significance, 1-3
 - units expressing, 3-2
- Thermal tab
 - continuous measurement mode parameters set in, 5-6–5-7
 - defining thermal measurement settings, 3-6
- Thermal Transport connection cable
 - connector ports, 2-6
 - function, 2-6, C-1
 - part number, 1-2
 - sample connections, C-2
 - in Thermal Transport connection diagram, A-2
- Thermal Transport control center
 - measurement menu, 3-5–3-8
 - opening, 3-3
 - showing system status, 3-9
 - tabs, 3-3–3-5
- Thermal Transport log
 - identifying calibration files, 3-2
 - recording errors, 3-14
 - recording messages, 3-9
 - viewing, 3-2
- Thermal transport measurements. *See* Measurements
- Thermal Transport option
 - activating, 5-3, A-3
 - connection diagram, A-2
 - data files. *See* Measurement data files; Raw data files
 - design benefits, 1-7
 - hardware, 2-1. *See also* Thermal Transport connection cable; Thermal Transport sample puck; User bridge board; User's kit; WaveROM EPROM
 - installing, A-1–A-3. *See also* Installation: Secondary maintenance. *See* Puck adjustment tool; Puck fingers measurements. *See* Measurements
 - overview, 1-1, 1-2
 - parameters, 1-2
 - pinouts, C-1–C-3
 - software. *See* Thermal Transport software
 - status codes, B-3
 - system requirements, 1-2
- Thermal transport properties
 - significance, 1-3
 - types measured, 1-5–1-6
- Thermal Transport sample puck
 - affecting sample size, 4-2
 - description, 2-2
 - illustration
 - with calibration fixture, 2-9
 - with puck-mounting station, 2-3
 - with radiation shield, 2-2
 - maintenance. *See* Puck adjustment tool; Puck fingers
 - mounting sample on, 5-3. *See also* Puck-mounting station
 - part number, 1-2
 - sample connections, C-2, C-3
- Thermal Transport software
 - control center. *See* Thermal Transport control center
 - data files. *See* Measurement data files; Raw data files
 - design versatility, 3-1
 - estimating errors, 1-9–1-10
 - installing, A-3
 - log file. *See* Thermal Transport log
 - models, 1-8–1-9
 - wizards. *See* Wizards
- Thermoelectric figure of merit. *See* Figure of merit
- Toolbox, containing user's kit, 2-4
- Troubleshooting
 - data, 6-1–6-3
 - High-Vacuum option, 6-3
- TTO. *See* Thermal Transport option
- Tto.ini file, 3-2
- Tto.Log.txt file, 3-9
- TTO Measure command, 3-1, 5-5
- Turbo Pump. *See* High-Vacuum option
- Two-probe lead configuration
 - minimizing resistance for, 4-1
 - suggested use, 4-4
- User bridge board, 2-7
- User's kit
 - contents, 2-3
 - illustration, 2-4
 - part number, 1-2
- Waveform tab, 3-4
- WaveROM EPROM
 - illustration, on AC board, 2-5
 - part number, 1-2
 - replacing, 2-6
 - upgraded version for Thermal Transport option, 2-1, 2-5
- Wizards
 - calibration, 2-9–2-10, 3-5
 - installation, 3-3, 5-3